EFFECTS OF PROCESS PARAMETERS ON PROPERTIES OF TRANSPARENT WOOD

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Certificates Project

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Department	Industrial engineering
Academic year	2016

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Abstract

This project studied effects of process parameters on properties of transparent wood. We used balsa wood because it is light, thin and can be cut easily. To make transparent wood, we followed three-step process. First, we first boiled it in a mixture of sodium hydroxide (NaOH) and sodium sulfite (Na_2SO_3) and then in hydrogen peroxide (H_2O_2) and filled in polyurethane polymer. The parameters that we varied were (1) the ratio of NaOH : Na_2SO_3 mixture and (2) ratio of polyurethane (PU): thinner. The results showed that the NaOH : Na_2SO_3 of 1:1 and 2:1 are the most suitable conditions to remove lignin. In terms of polymer filling, the ratio of PU : thinner of 50:50 is the best for 1:1 condition and the ratio of PU : thinner of 75:25 is the best for 2:1 condition. It was also found that the strength of transparent wood lower than that of the original wood.

Acknowledgment

This project cannot be accomplished without the help of many people. We especially thank Dr.Narumon Seeponkai, Dr.Duong Au Thanh Binh, Dr.Jirapat Ananpattarachai, our advisor and co – advisors, for their time, advices and suggestions throughout the project.

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Project group

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May, 2017

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Contents Symbols and Abbreviations

NaOH	=	Sodium hydroxide
Na_2SO_3	=	Sodium sulfite
H_2O_2	=	Hydrogen peroxide
H_2SO_4	=	Sulfuric acid
g	=	Gram
cm	=	Centimeter
MPa	=	Megapascal
^{o}C	=	Degree Celsius
min	=	Minute
ml	=	Milliliter
M	=	Molar
%	=	Percent
N	=	Newton
mm	=	Millimeter
etc.	=	Et Cetera
hr./hrs.	=	Hours

Chapter 1

Introduction

1.1 Statement of the problems

Currently, transparent products are made of glasses or polymers, which are heavy, fragile or require many chemicals in the process – resulting in hazardous wastes. Compared to glasses and polymers, woods have better mechanical properties, long lifetime and can be naturally degraded. If woods can be made to become transparent, it will create alternative transparent products which can be used in constructions, storage, and other applications where mechanical strength and optical appearance are both important. In this project, we proposed to design a process to make transparent woods and study effects of process parameters on properties of transparent woods.

1.2 Objective

1.2.1 To analyze the effects process parameters of produce transparent wood (geometry chemical)

1.2.2 To optimize process parameters of produce transparent wood

1.3 Project output

At the end of this project transparent wood will be obtained

1.4 Project outcome

Products made of transparent wood achieve 80 % transparency

1.5 The scope of project

1.5.1 One types of woods (Balsa wood) was studied

1.5.2 Wood thickness of 0.1 cm was used

1.5.3 Effects of chemical concentrations on properties of transparent wood will be studied

1.6 The place of project

This project be was carried out in Faculty of Engineering at Naresuan University

1.7 The time of project

This project was studied from August 2016 to April 2017

1.8 Timeline

Table 1 Procedures and plan

Sequence	Progress	Aug.	Sep.	Oct.	Nov.	Dec.	Jan.	Feb.	Mar.	Apr.
1	Literature review	↔								
2	Design experiment		↔							
3	Investigate: Chemical concentration			←	—	—	→			
4	Optimization							↔		
5	Summary and report								←	→

Chapter 2

Literature review

In order to study effects of process parameters on properties of transparent wood, it is necessary to understand the basic of wood, as follows.

2.1 Tree

A tree has two main parts, the roots and the shoot. The roots respond for water uptake, providing mechanical support of the shoot and storing nutrition. The shoots consist of trunk, branches and leaves. Trunk is the main part that is used for making wood products. From here on, the trunk part of the tree will be discussed.

Softwood vs. Hardwood

According to botanical definition, softwoods are woods of trees with needle-leaved trees such as rubber wood, bamboo wood, pinewood, etc. They are lightweight and easy process. Hardwoods are woods from broadleaf trees such as maple, teak wood, makah wood, takien wood, etc. They are heavy weight and durable. Softwoods and hardwoods are also different in terms of their cells. Cells in hardwoods have vessel element where softwoods lack these.

2.2 Structure of wood

The trunk of both softwoods and hardwoods consists of three zones: heartwood, sapwood and bark (Figure. 2.1 [1]). Heartwood zone is the dark colored wood found near center of the trunk. Heartwood is the innermost zone of the trunk, where long-term biochemical storage is kept. Sapwood zone is the band with lighter color in between the heartwood zone and the bark. The sapwood is responsible for transporting water and minerals (sap), and also synthesizing and storing nutrition for photosynthesis process.

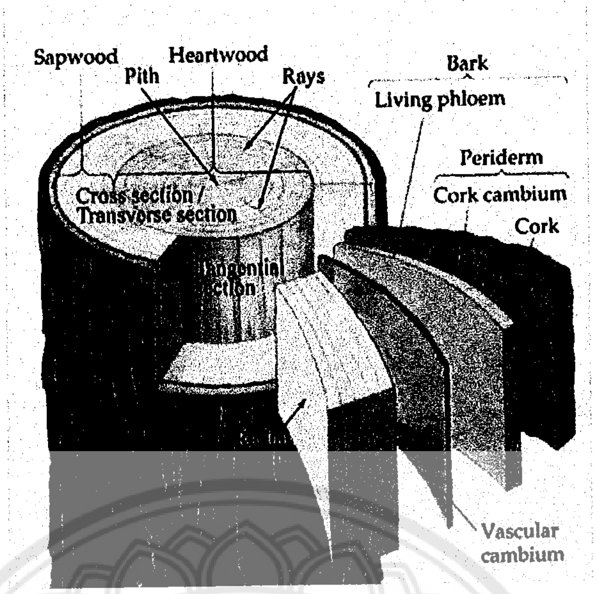


Figure 2.1 A cut-through of a tree trunk [1]

The bark zone is the outmost layer of the trunk, consisting of mostly dead cells, yielding its dark color on the outside of the trunk. The bark is responsible for protecting the inner zones from environmental damages.

Along the longitudinal direction, the structure of wood is quite different than radial direction. Long aligned vertical channels are observed in the longitudinal section. They are used to pump water and ions up from the root. The hierarchical structure and strong bonds between wood cells give excellent mechanical properties in woods.

2.3 Wood compositions

The composition of wood depends on the type of tree. In general, wood cell wall has around 40-50% cellulose microfibrils, 25-30 % hemicellulose, 15-25% lignin and 5-10 % others as shown Figure 2.2 [2].

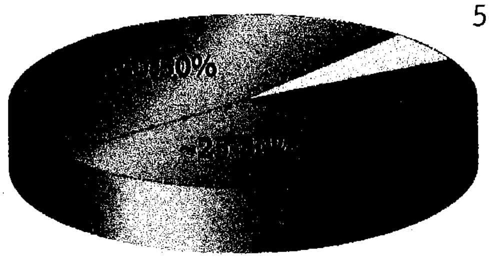


Figure 2.2 Lignocellulosic biomass in wood [2]

2.3.1 Cellulose and Hemicellulose

Cellulose is a natural polymer and the important ingredient of the wood. Due to strong bond between cellulose making it look like fiber, people often call it cellulose fibrils. Hemicellulose is made of carbohydrate polysaccharide type molecules called heteropolysaccharide. Hemicellulose is responsible for storing water and ion exchange. Both cellulose and hemicellulose are optically colorless.

2.3.2 Lignin

The amount of lignin in normal wood is 15-25% depending on the different wood species. Lignin is made of high molecular weight cross-linked phenolic polymers. Lignin is very durable and serve as "glue" filling the spaces between the cell walls. It is covalently bonded with cellulose and hemicellulose, giving mechanical strength to the cell wall. While cellulose, hemicellulose and other polysaccharides on cell walls are hydrophilic, lignin is hydrophobic. Lignin has a complex chemical compound with dark color as shown Figure 2.3 [3].

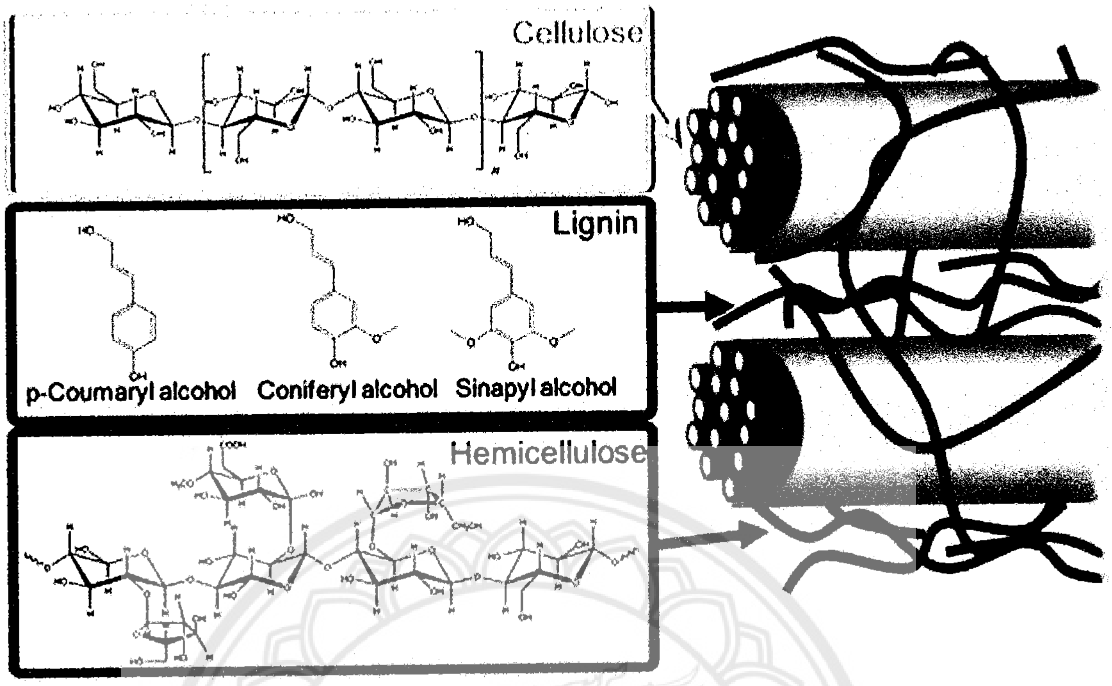


Figure 2.3 The composition of wood [3]

The hierarchical structure of wood and color generated from complex lignin compound cause light scattering in the visible range. This creates different non-transparent colors in woods.

2.4 Delignification

Wood can be made transparent through chemical bleaching process as in paper production. This process is usually referred as kraft process or delignification process. This process consists of five steps as shown Figure 2.4 [4].

2.4.1 Impregnation

The woods is cut into chips, and boiled in a mixture of water, sodium hydroxide and sodium sulfide to break bonds between lignin and cellulose and hemicellulose.

2.4.2 Cooking

The wood chips are transferred to pressurized vessels called digesters to continue break lignin and hemicellulose into fragments.

2.4.3 Screening

the wood chips are then collected and passed through different sieves to remove dirt and debris. The product at the end of screening process is called wood pulp.

2.4.4 Bleaching

Bleaching is the chemical process to further decrease the lignin content in the pulp to make paper becomes whiter. Typically, pulp would be treated further with hydrogen peroxide and sodium dithionite to deprotonate lignin phenolic groups.

2.4.5 Washing

The pulp is finally washed with water to remove chemical residue and squeezed through rollers to remove water. After that the paper would be taken through hot steamrollers until the remaining water is very low.

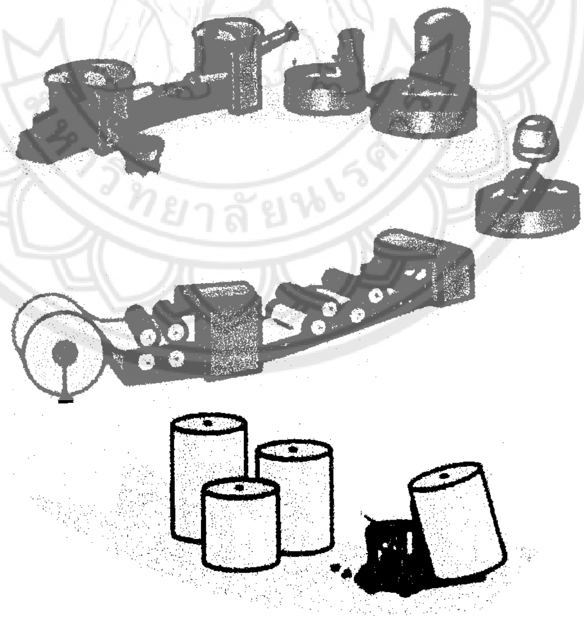


Figure 2.4 Paper making process [4]

2.5 Mechanical evaluation

Mechanical properties of materials are required for their potential applications. Same as other materials, mechanical properties of wood is evaluated using tensile tests. The samples are cut into standard size and subjected to a controlled applied load until failure. During the testing process, the internal resistance of the material's structure, with the external pressure is measured. Stress can be calculated as shown in equation 2.1

$$\sigma = F/A \qquad (2.1)$$

where: σ = Stress due to pull the work piece (MPa)

F = Maximum force is used to pull (N)

A = Cross-sectional area of the specimen (mm2)

When external force is applied on the material, it not only causes the resistance of internal force of the material only but also the material deformation. This means the material is deformed under the applied load. The deformation or strain can be calculated using equation 2.2

$$\varepsilon = \Delta L/L\_0 \qquad (2.2)$$

where: ε = Strain

ΔL = Length changes (m)

L_0 = Original length (m)

Based on the stress-strain measurements obtained from the tensile test, other properties such as elastic modulus, yield strength, etc. can also be determined.

Furthermore, observation fracture morphology, cause of failure can be determined and prevented as shown in Figure 2.5 [5] and 2.6 [5].

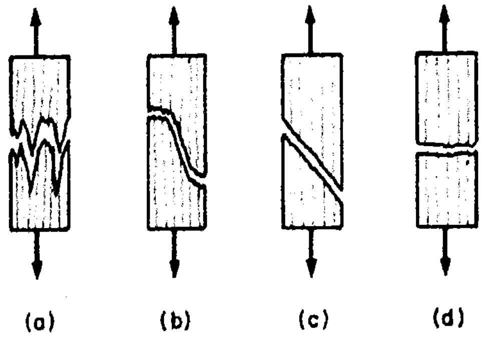


Figure 2.5 Fracture morphology (a) splintering tension, (b) combined tension and shear, (c) shear, (d) brittle tension [5]

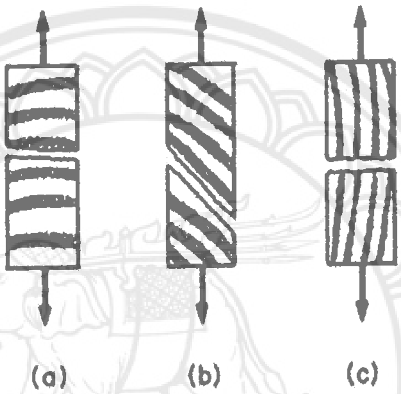


Figure 2.6 Fracture morphology (a) tension failure of early wood, (b) shearing along a growth ring, (c) tension failure of wood rays [5]

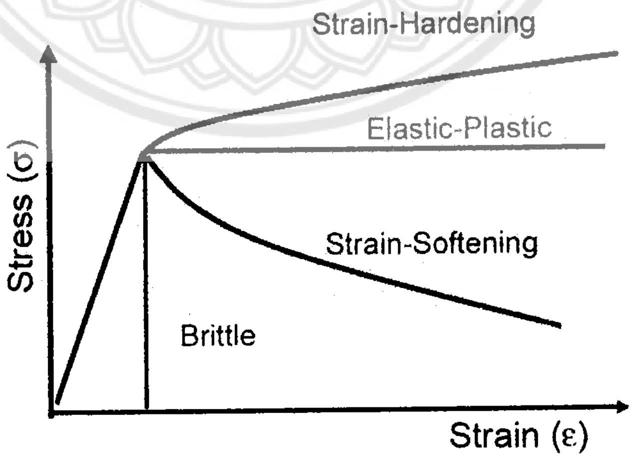


Figure 2.7 The relationship between the stress - strain [6]

2.6 Optical properties

Optical properties can be check using a lux meter. Lux meter is used to measure the amount of light and light intensity transmitted through an object. Lux meter has unit of lux and foot candle. Lux describes the photon flux per unit area, which is equivalent to one lumen per square meter. The photodetector equipped inside the lux meter is placed perpendicular to the light source to obtain the measurement.

Figure 2.8 Lux meter for measure light [10]

2.7 Related research

Mingwei Zhu et al (2016), studied the anisotropic effects of woods on optical and mechanical properties of transparent woods. These wood composites are highly transparent, with a decay rate of no more than 90%, but with varying optical and mechanical properties. Compared with the nature L-wood, transparent L-wood after polymer infiltration has a higher strength and ductility. The transparent L-wood also has a higher ductility than the transparent R-wood. The R-wood transmittance is up to 90% higher than L-wood.

Yuanyuan Li et al (2016), studied the optically transparent wood was fabricated by bulk infiltration of refractive-index-matched, pre-polymerized methyl methacrylate (MMA). High optical transmittance of 85% and haze of 71% were achieved at a transparent wood thickness of 1.2 mm. Optical properties of transparent wood are tunable by changing the cellulose volume fraction.

Chapter 3

Research methodology

In this project, our group investigated effects of chemicals concentration and polymer resin fillers on the mechanical and optical properties of transparent woods.

Experimental Procedure

The process of making transparent wood

Wood was cut size 2 cm x 2 cm. It was soaked in a boiled mixture of NaOH and Na_2SO_3 (Table 3.1) at 80°C for 4 hrs. It was then rinsed with hot distilled water three times to remove the chemicals before soaking it again in a H_2O_2 solution to remove excess lignin. Wood was rinsed with cold water. The wood finished product was preserved in ethanol before filling it with polymer resin according to the diagram showing the experimental procedure below.

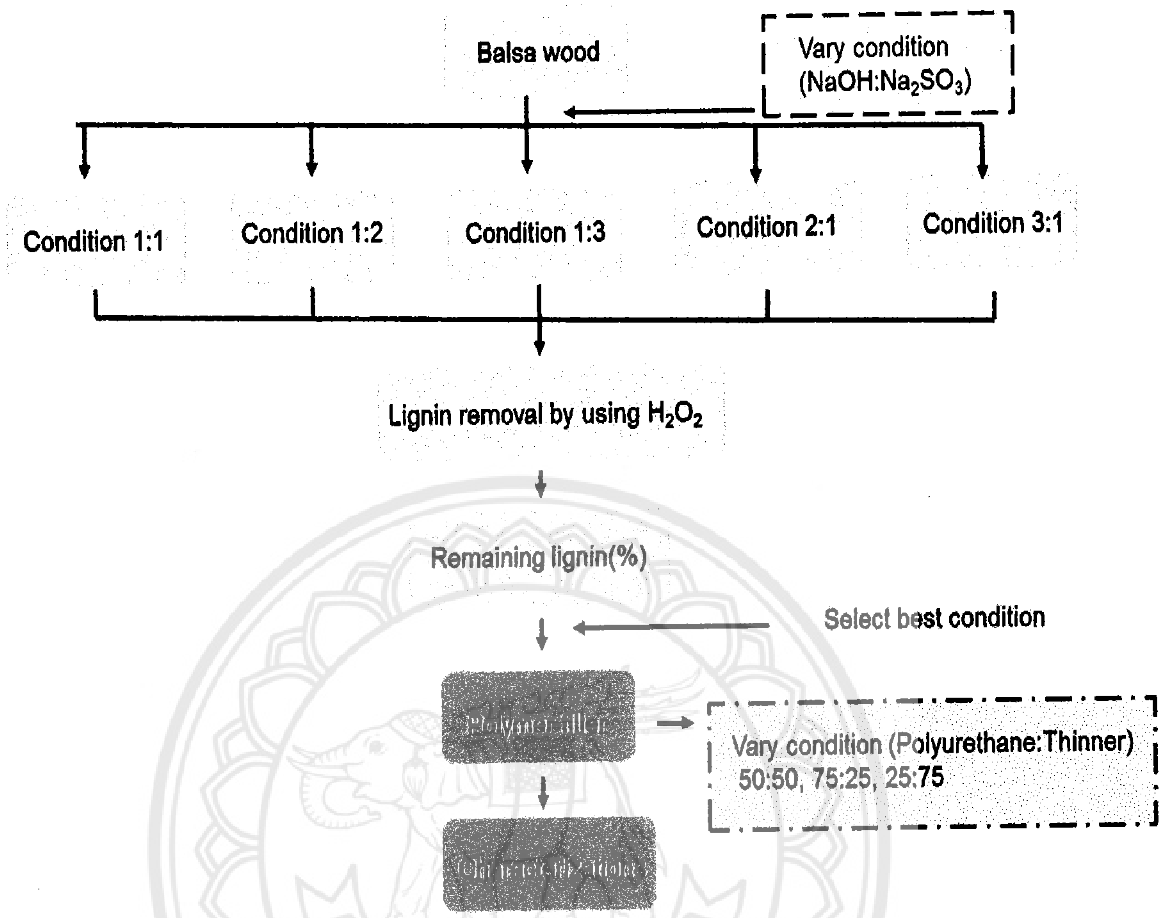


Figure 3.1 The diagram shows an experimental procedure

3.1 Effects of chemicals on lignin removal

In this project, we compared the lignin content in wood after the two lignin-removal processes to provide an optimum process. In process 1, part of the lignin was removed in the boiled mixture of $NaOH:Na_2SO_3$. We varied the ratio of the two chemicals according to Table 3.1 and Table 3.2 below. In process 2, the remaining lignin was dissolved in H_2O_2 solution at concentrations 5.0 M.

Table 3.1 Use solution 100 ml of $NaOH:Na_2SO_3$

$NaOH:Na_2SO_3$(Use solution 100ml)					
$NaOH(mol):Na_2SO_3(mol)$	1:1	1:2	1:3	2:1	3:1
$NaOH(g):Na_2SO_3(g)$	4:12.6	4:12.6	4:12.6	8:12.6	12:12.6
Time(hr)	4				

Table 3.2 Use solution 50ml of $NaOH:Na_2SO_3$

$NaOH:Na_2SO_3$(Use solution 50ml)					
$NaOH(mol):Na_2SO_3(mol)$	1:1	1:2	1:3	2:1	3:1
$NaOH(g):Na_2SO_3(g)$	2:6.3	2:12.6	2:18.9	4:6.3	6:6.3
Time(hr)	4				

3.2 Effects of polymer fillers

Although colored lignin in wood is removed, delignified wood is still not transparent due to light scattering at the interface between cell wall and air. To achieve high optical transparency, we filled in a polymer resin that has refractive index close to that of cell wall. The refractive indices of lignin and cellulose are 1.61 and 1.53, respectively. In this study, we used polyurethane (refractive index: 1.50). Commercially available polyurethane is a highly viscous liquid and requires a thinner solution to dilute for practical use. We studied effects of different mixing ratio between commercial polyurethane and thinner solutions (Polyurethane: thinner volume ratio – 50:50, 25:75, 75:25)

3.3 Characterization

3.3.1 Remaining lignin (%) (Solution test)

The content of lignin removal was determined following Technical Association of Pulp and Paper Industry Standard Method T 222-om-83. Briefly, 1g of dry wood (M) is measured and extracted with ethanol for 4 hours, and then treated with cold sulfuric acid solution (72%, 15mL) for 2 hours under stirring at 20°C. Then, 560mL distilled water is added to dilute sulfuric acid to 3%. The solution is continued boiled for 4 hours. The solution is cooled down, filtered, washed with distilled water and dried. The insoluble materials is weighed (m). The lignin content can be calculated as:

$$\text{Lignin, \%} = \frac{m}{M} \times 100\% \quad (3.1)$$

Where:

m = Mass of lignin, g

M = oven-dry mass of test specimen, g

3.3.2 Tensile Test

The transparent wood products was cut to standard test dimensions of width 3 cm and length 10 cm thickness 0.1 cm. Mechanical properties of the transparent wood was tested using universal testing machine. Stress-strain curves obtained from the test was analyzed and compared between different samples by giving size of Figure 3.1.

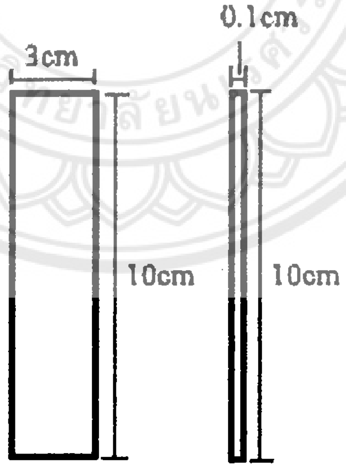


Figure 3.2 Wood used Tensile Test

3.3.3 Measure transparency test

A model house with dimensions of 12cm x 15 cm x 15cm was built. The light bulb was on top. Transparency inside the model house was measured using lux meter.

Chapter 4

Experimental results and Analysis

This chapter discusses experimental results and analyzes experimental results. First, we identified the best chemical conditions to remove lignin, and found out the best polyurethane:thinner ratio to fill in the delignined-woods. After that, we performed mechanical tests and analyzed the light transmittance of the transparent woods.

4.1 Effects of NaOH : Na_2SO_3 ratio

Figure 4.1 shows the percentage of remained lignin in the wood after the two chemical treatment steps. It clearly shows that the minimum lignin of 0.69% is remained when 2 NaOH: 1 Na_2SO_3 and 5M H_2O_2 are used. The percentage of remaining lignin after the first chemical treatment step for 1:1, 1:2, 1:3, 2:1 and 3:1 conditions are 5.4%, 4.31%, 4.31%, 4.41% and 4.95%, respectively. The amount of lignin is substantially removed after the second step of chemical treatment in boiled H_2O_2, 0.69% and 0.99%, respectively. Because the remaining lignin after two step treatments of 1:1 and 2:1 ratios give similar values of about 0.8%, we decided to use de-ligninfied wood at these conditions for filling in polyurethane.

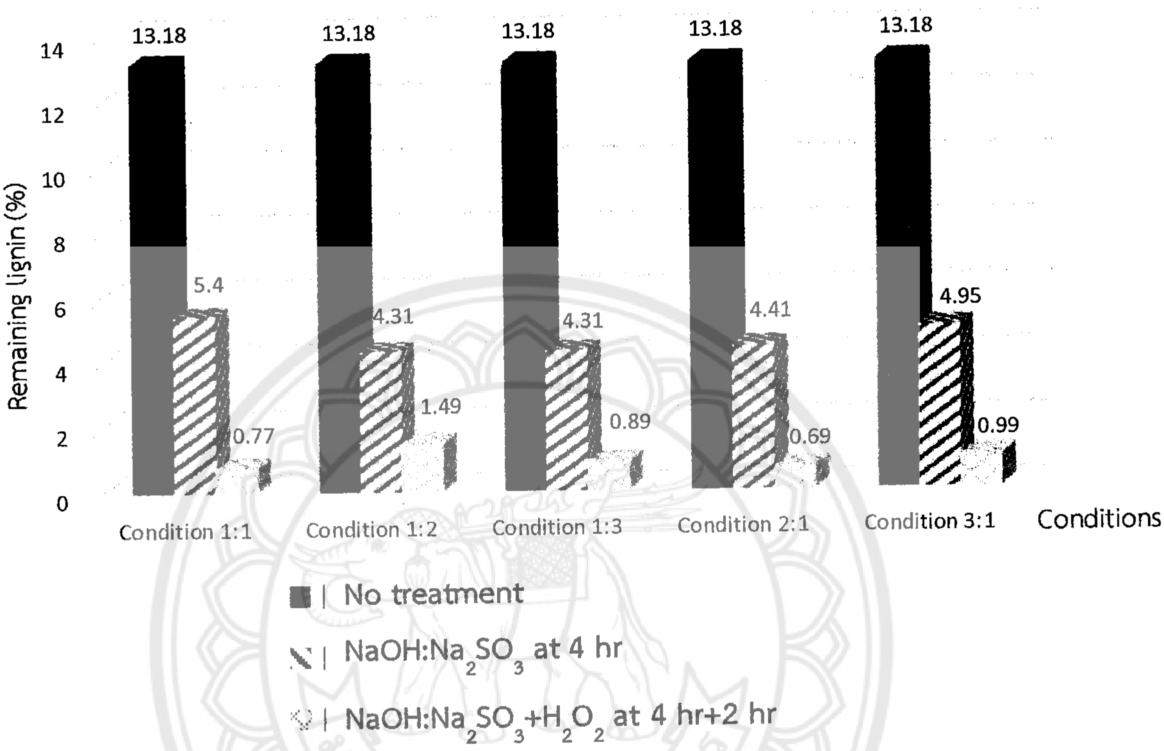


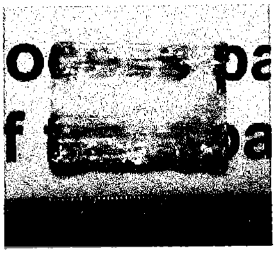
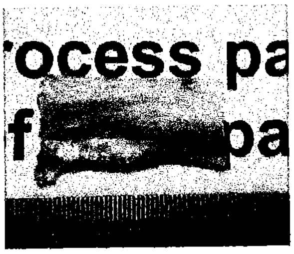
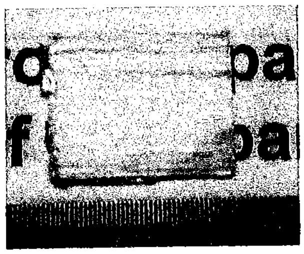
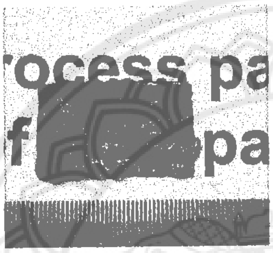
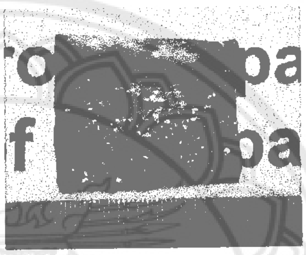
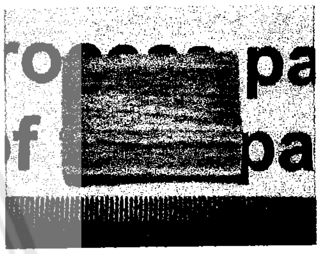
Figure 4.1 Percentage of remaining lignin after treated at different NaOH : Na_2SO_3 ratio

4.2 Effects of polymer filler

Polyurethane: thinner ratios of 50:50, 75:25, 25:75 were used to fill in deligninfied wood treated by 1:1 and 2:1 ratios. The polymer filled woods are shown in Table 4.1.

Based on the visibility of letters behind the wood, it clearly shows that polyurethane:thinner ratio of 75:25 is best for filling deligninfied wood of 2:1 condition. For deligninfied wood at 1:1 condition, the visibility is not sufficient.

Table 4.1 Photographs showing de-ligninfied woods after filling with polyurethane

Condition	50:50	75:25	25:75
1:1 Dry			
2:1 Dry			

4.3 Mechanical analysis of original wood and transparent woods

4.3.1 Analyze tensile test

The mechanical property of transparent woods was tested using universal testing machine at a speed of 21 mm/min and force 5 N. Original woods have gauge length of 30 mm x 80 mm and transparent woods have gauge length 15mm x 64 mm.

Figure 4.2 shows the strength of original woods and transparent woods. The original wood strength has values as 25.2 (MPa), while the strength of the transparent woods at 1:1 condition and 2:1 condition are 24.9 MPa and 21.1 MPa, respectively.

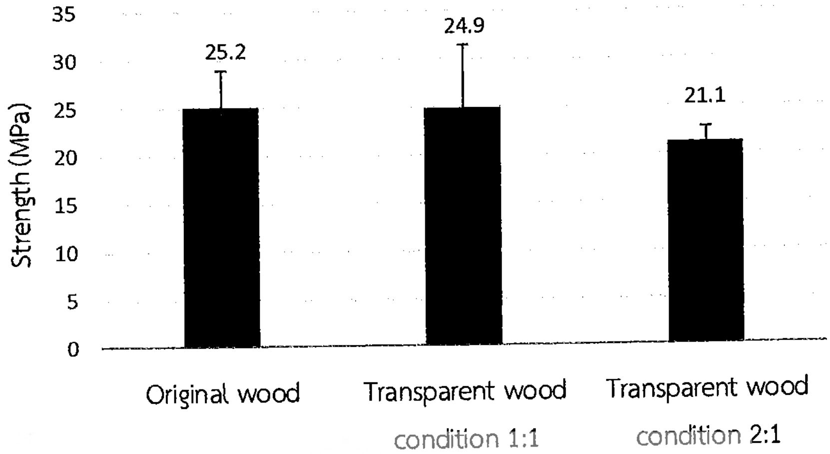


Figure 4.2 Strength of original wood and transparent woods at different conditions

The Young's modulus values of different woods are derived from the stress-strain curve obtained from tensile test as shown in Figure 4.3. The original wood, transparent wood condition 1:1 and transparent wood condition 2:1 Young's modulus has values as 3,217, 451 and 504 MPa, respectively. The results implies that the original wood has higher stiffness than the transparent wood, meaning the original wood resists to the change of length or there is a slight change in shape when stress is applied.

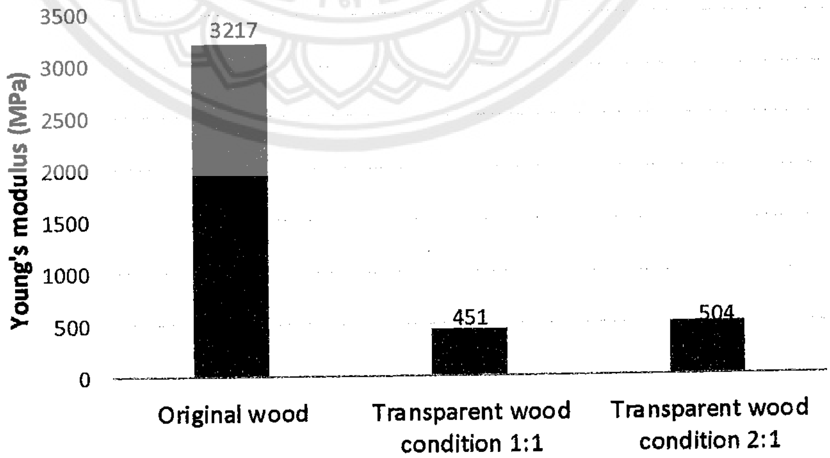


Figure 4.3 Young's modulus of original wood and transparent woods at different conditions

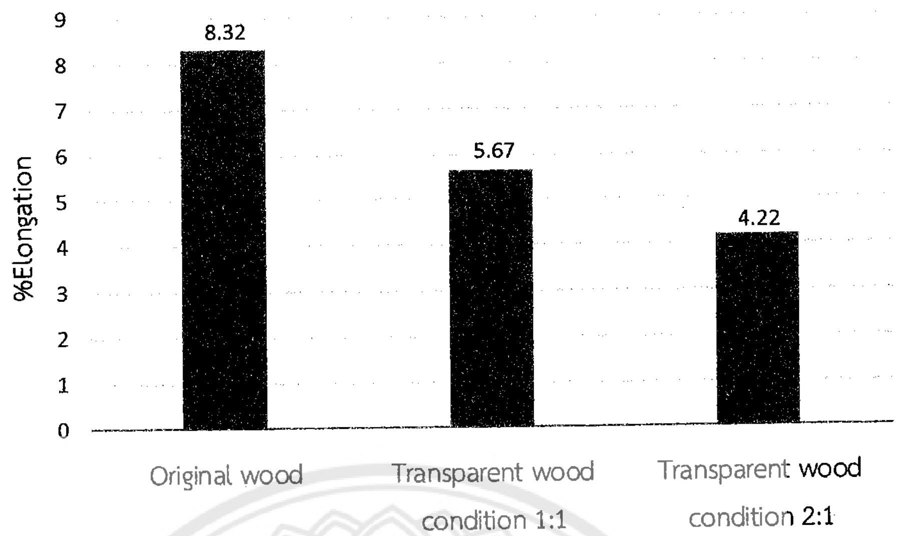


Figure 4.4 Ductility of original wood and transparent woods at different conditions

Figure 4.4 shows dutility of the original wood, transparent wood condition at 1:1 and transparent wood condition at 2:1. %Elongation values are 8.32, 5.67 and 4.22 (MPa) for original wood, transparent wood at 1:1 condition and 2:1 condition, respectively. The results indicate that original wood is more ductile than the transparent woods.

4.3.2 Transparency analysis

For transparency measurement, the distance between transparent wood and lamp was set at 7 cm. The distance between transparent wood and lux meter was 7.5 cm. and house design shown in Figure 4.6.

Figure 4.5 Wood house for measured lux meter

The two types of transparent woods were measured using lux meter. The transparent woods at condition 1:1 and condition 2:1 measured have the values 33.2 lux and 59.4 lux, respectively, indicating condition 2:1 can transmit more light then condition 1:1. shown in Figure 4.6.

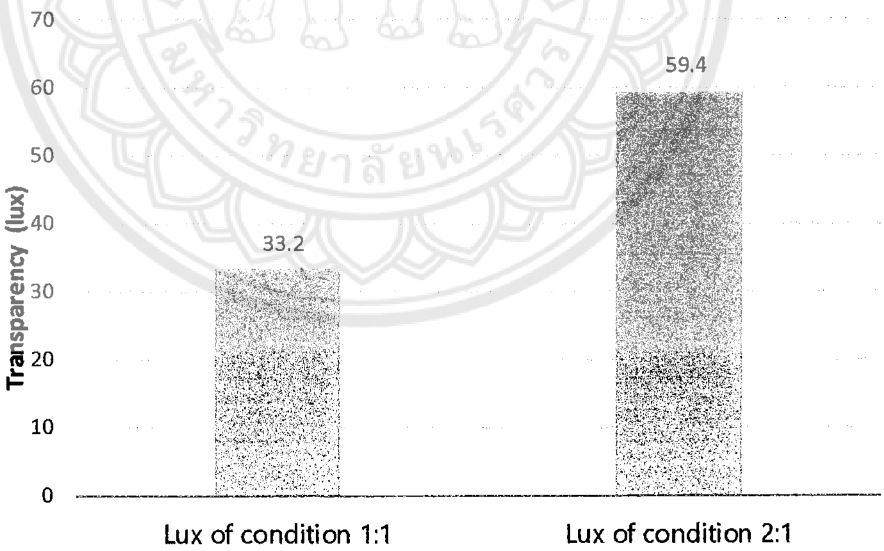


Figure 4.6 Compare the values of transparency

Chapter 5
Conclusions and Recommendations

5.1 Conclusions

In this project, we studied the process of making transparent wood and effects of chemical treatment on the amount of lignin removal of balsa wood. We found that treating woods first in boiled 2M NaOH : 1M Na_2SO_3 for 4 hours and then for 2 hours in 5M H_2O_2 yields 0.69% lignin remaining. To obtain transparent wood, the deligninfied wood should be filled with polyurethane : thinner at 75:25 ratio. After measuring tensile strength, original wood is not fracture but transparent wood condition 1:1 and condition 2:1 did fracture. The transparent wood condition 1:1 fracture more condition 2:1 show that condition 2:1 very strength, Young's modulus condition 2:1 more condition 1:1 but ductility condition 1:1 more condition 2:1. After measuring lux meter. The transparent wood condition 2:1 more valuable condition 1:1 for lux, show that condition 2:1 was transparent well.

5.2 Recommendations

Although this project has completed, further studies is still needed. It would be very interesting to study more on how types of wood, wood thickness, types of polymer filler affect the mechanical and optical properties of the transparent woods.

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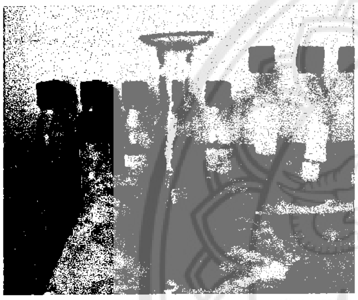
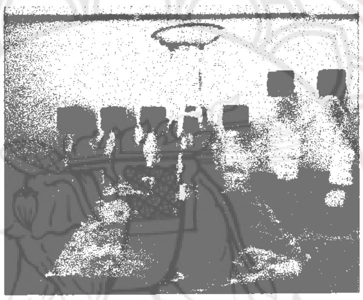
Appendix A

Step1. Boil in NaOH withNa_2SO_3

Step2. Boil in H_2O_2

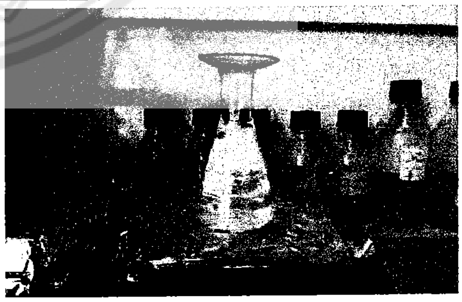
Step1. Boil in NaOH with Na_2SO_3

Table A1 Experiment boil in NaOH with Na_2SO_3 for 4 hr

Start	Time passes 1 Hour	Time passes 2 Hour
		
Time passes 3Hour	Timepasses4 Hour	
		

Step2. Boil in H_2O_2 for unit wood becomes white

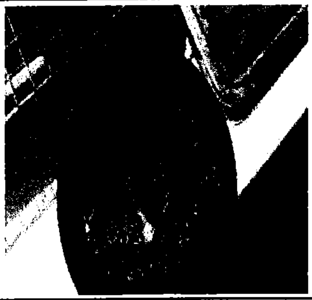
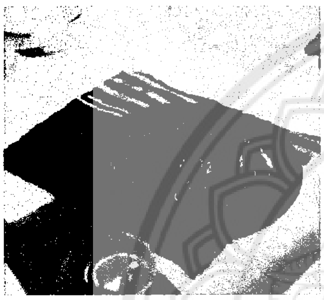
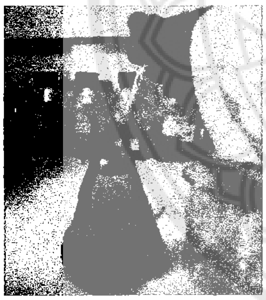
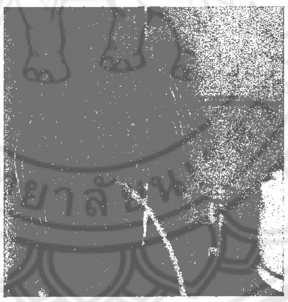
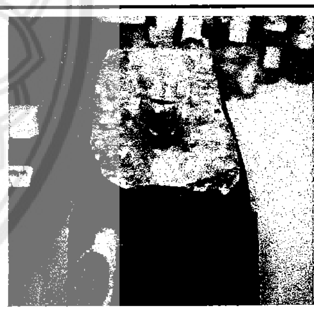
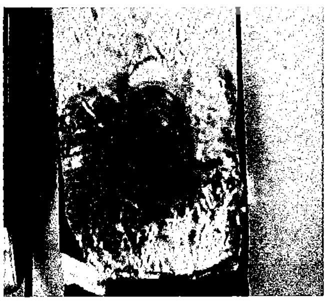
Table A2 Figure of experiment boil in H_2O_2 for unit wood becomes white

Start	Time passes unit wood becomes white
	

Appendix B

Remaining lignin (%)

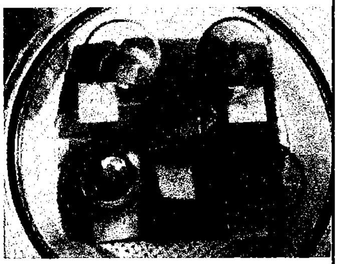
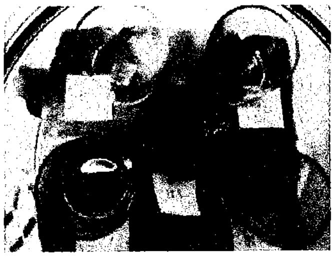
Table B Experiment remaining lignin (%)

Weigh	Add ice	Put wood
		
Add sulfuric acid	Control 20 degree	Boiled 4 hr.
		
Out sulfuric acid	Filter lignin	Dry oven
		
Weigh for calculate		
		

Appendix C

Polymer filler

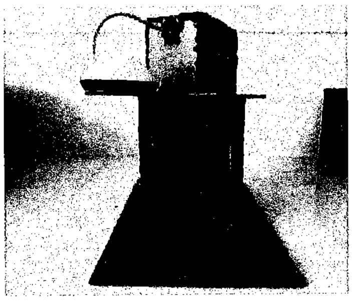
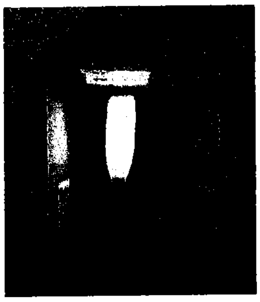
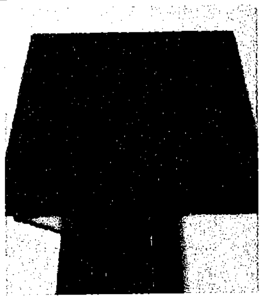
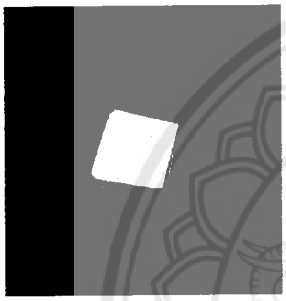
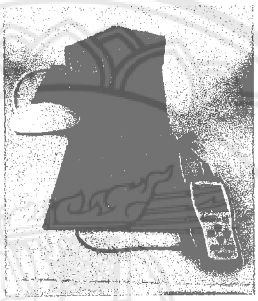
Table C Experiment polymer filler

Mix polyurethane: thinner	Start wax	Wax 5 times
		

Appendix D

Transparency and temperature test

Table D Figure of experiment transparency and temperature test

Wood house	The top	Place wood
		
Light through wood	Use lux meter measure	
		

Appendix E

Tensile test

Table E1 Experiment tensile test

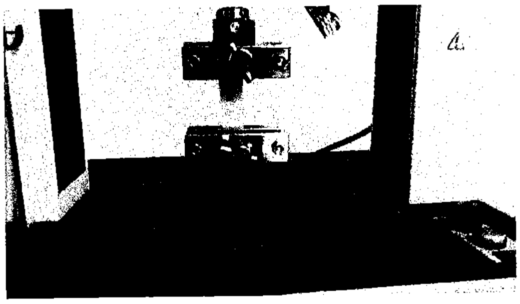
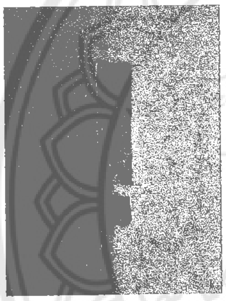
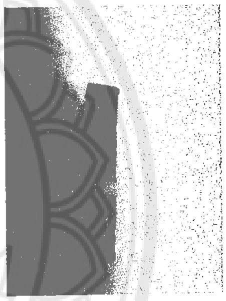
Original wood	Transparent wood
	

Table E2 Result of tensile test

Transparent wood condition 1:1	Transparent wood condition 2:1
	

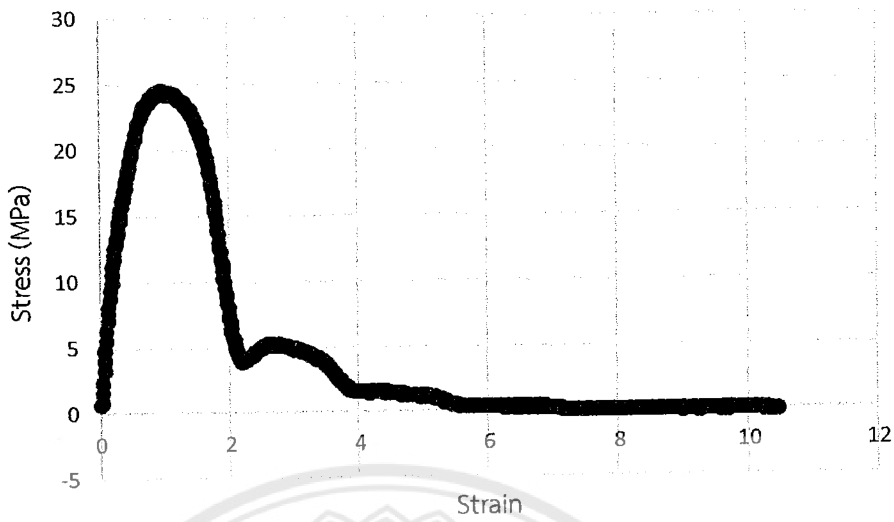


Figure E.1 Tensile test for stress-strain curves of original wood (1)

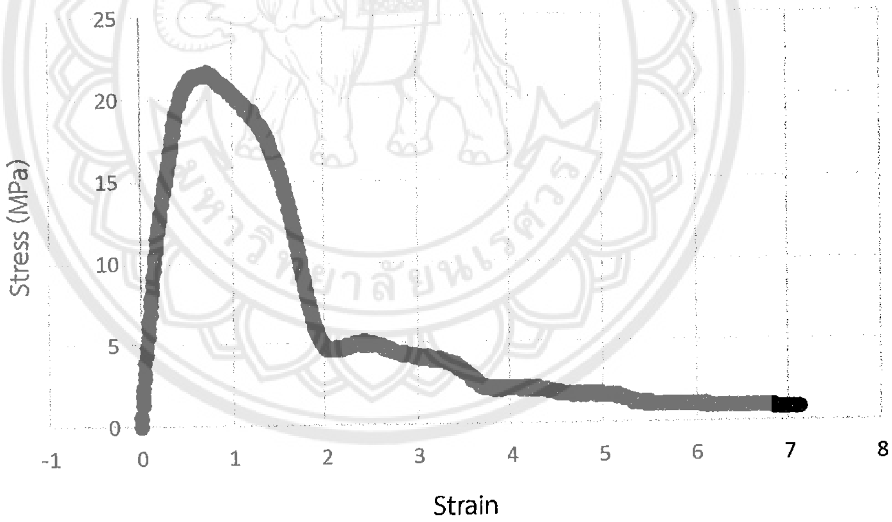


Figure E.2 Tensile test for stress-strain curves of original wood (2)

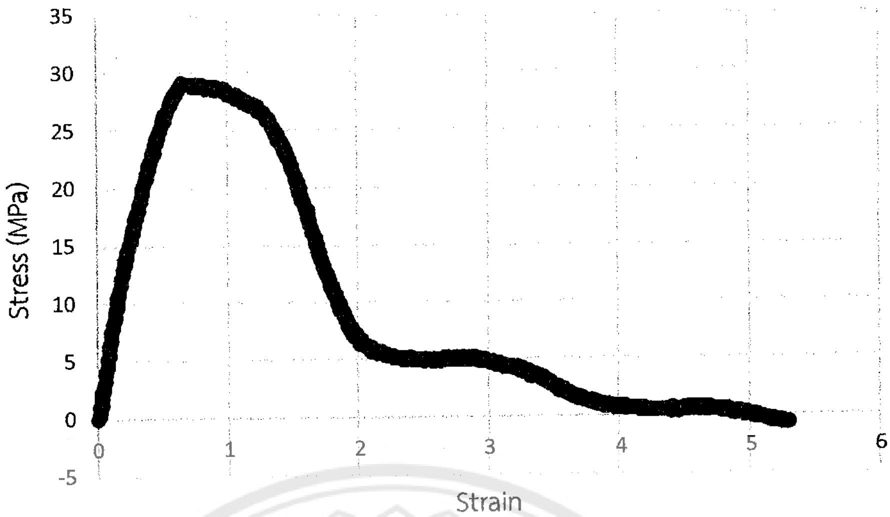


Figure E.3 Tensile test for stress-strain curves of original wood (3)

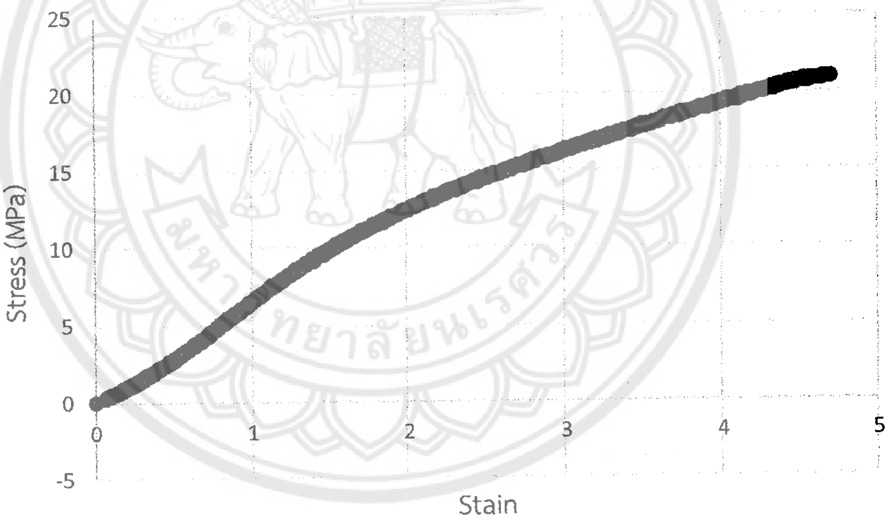


Figure E.4 Tensile test for stress-strain curves of transparent wood condition 1:1 (1)

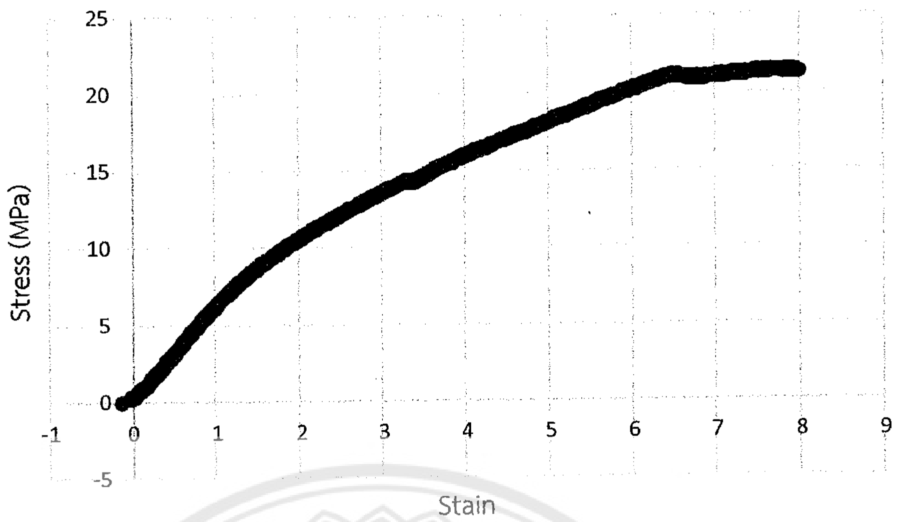


Figure E.5 Tensile test for stress-strain curves of transparent wood condition 1:1 (2)

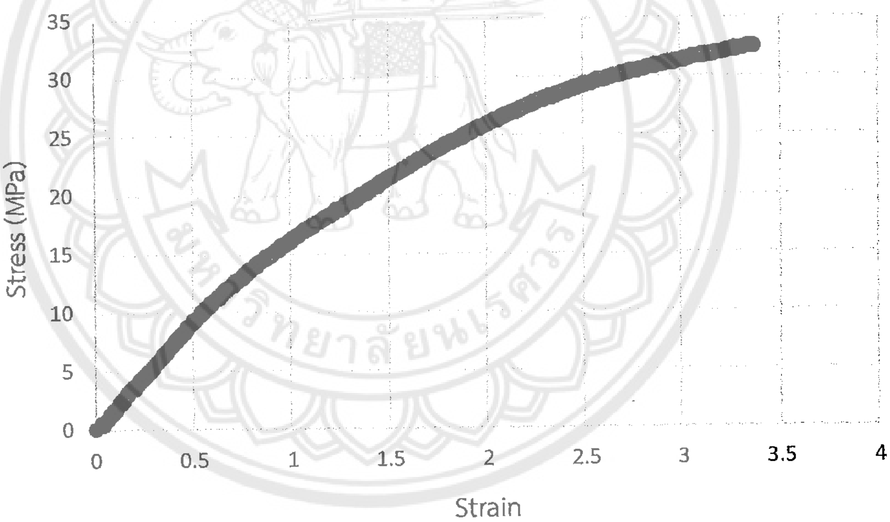


Figure E.6 Tensile test for stress-strain curves of transparent wood condition 1:1 (3)

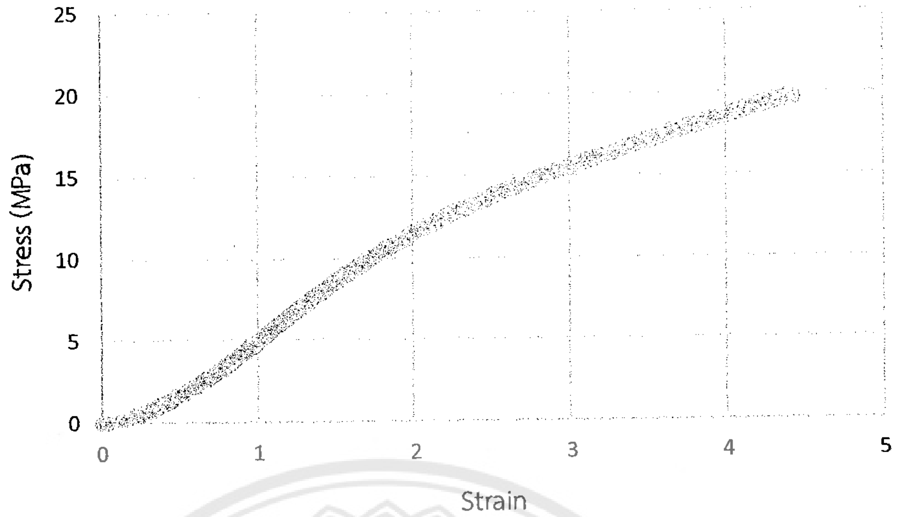


Figure E.7 Tensile test for stress-strain curves of transparent wood condition 2:1 (1)

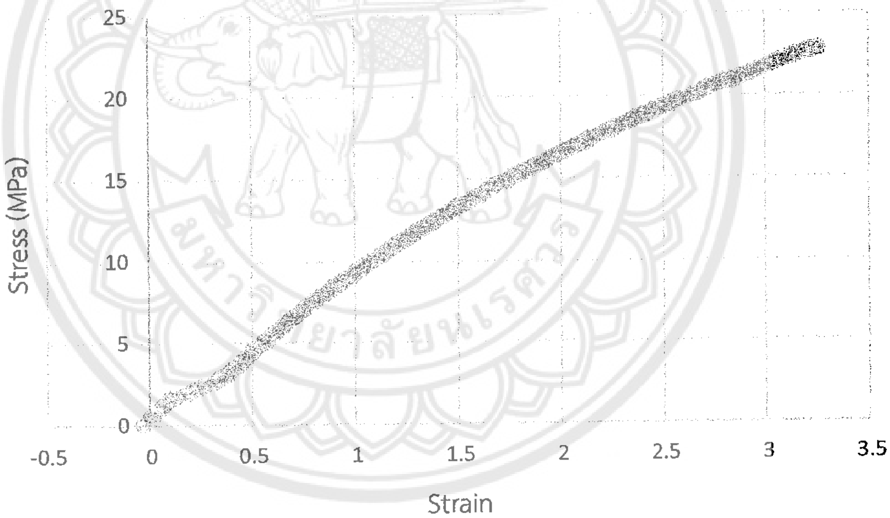


Figure E.8 Tensile test for stress-strain curves of transparent wood condition 2:1 (2)

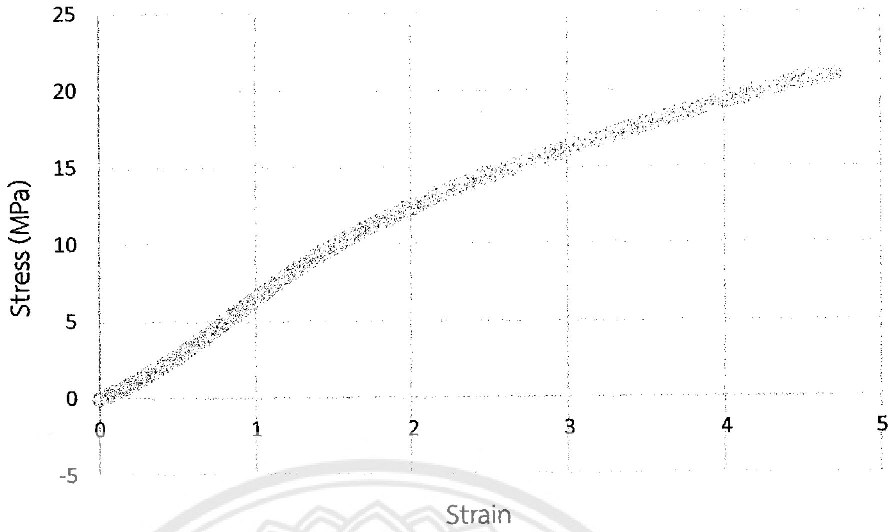


Figure E.9 Tensile test for stress-strain curves of transparent wood condition 2:1 (3)