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**Effect of Furfurylation Treatment on Technological Properties of Short rotation Teak Wood**

Resa Martha<sup>a,b</sup>, Mahdi Mubarak<sup>a,b</sup>, Irmanida Batubara<sup>c</sup>, Istie S. Rahayu<sup>a</sup>, Loedy Setiono<sup>d</sup>, Wayan Darmawan<sup>a\*</sup>, Firmin Obounou Akong<sup>b</sup>, Béatrice George<sup>b</sup>, Christine Gérardin<sup>b</sup>, Philippe Gérardin<sup>b\*</sup>

<sup>a</sup>Department of Forest Products, Faculty of Forestry and Environment, IPB University, Bogor 16680, Indonesia.

<sup>b</sup>Université de Lorraine, INRAE, LERMAB, F-54000 Nancy, France.

<sup>c</sup> Department of Chemistry, Faculty of Mathematics and Natural Sciences; and Tropical Biopharmaca Research Center IPB University, Bogor 16680, Indonesia.

<sup>d</sup>Researcher at PERHUTANI Research and Development Centre, Cepu 58313, Indonesia.

\*Corresponding authors email: wayandar@indo.net.id and philippe.gerardin@univ-lorraine.fr

## Abstract

Short rotation teak wood has low quality especially in dimensional stability and durability. Furfurylation treatment was applied to improve technological properties of the short rotation teak wood. The purpose of the study was to improve dimensional stability and durability through furfurylation treatment. Short rotation teak sapwoods (Plus Perhutani and Biotrop teaks) were treated through the impregnation process of 45% of furfuryl alcohol (FA) and 5% of tartaric acid as catalyst followed by heated in oven at 120 °C for 16 h under nitrogen atmosphere. Dimensional stability was characterized by volumetric swelling, anti-swelling efficiency (ASE), water uptake (WU), and bulking effect (BE), meanwhile decay durability was determined by the weight loss (WL). The results show that ASE increased in the average of 64%, WU decreased in the average of 59%, and BE increased in the average of 11%, which indicated the improvement of dimensional stability. Weight losses of furfurylated teak woods due to fungal decay of *Coriolus versicolor* (CV), *Pycnoporus sanguineus* (PS), and *Coniophora puteana* (CP) were in the average of 2%, 1%, and 1%, respectively, which categorized their durability to be class 1 (very durable). The increase of lignin content showed by Klason lignin content measurement and Fourier Transform Infrared (FTIR) and Carbon 13 Nuclear Magnetic Resonance (<sup>13</sup>C NMR) analysis indicated that the reaction of FA with lignin occurred after furfurylation treatment. Furfurylated sapwood presented also better thermic stability compared to untreated wood by Thermogravimetric Analysis (TGA). The FA treatment can be applied for improving the quality of short rotation teaks for high quality wood product utilization.

Keywords: dimensional stability, decay durability, FTIR and TGA analysis, furfuryl alcohol, lignin content, short rotation teak

## 1. Introduction

The supply of long rotation teak wood (*Tectona grandis* Linn. F.) has been declining year after year. The amount of long rotation teak wood production in Indonesia in 2017, the first quarter was 134500 m<sup>3</sup> and decreased in the fourth quarter to 115841 m<sup>3</sup> [1]. Short rotation teak is one of the solutions to overcome the limited supply of long rotation teak wood. Short rotation teak is known by various trade name, such as Biotrop teak is the short rotation teak planted by SEAMEO Biotrop and Plus Perhutani Teak is developed by the Perhutani Research and Development Center. Short rotation teak has several advantages such as short cutting cycle (7-15 years), lesser branches, straight and cylindrical trunk. However, short rotation teak wood has low strength, density and durability, and high proportion of juvenile wood [2,3]. Juvenile wood has shorter fibres with thinner walls, larger microfibril angles, lower densities, and lower strength properties compared to mature wood [4,5,6]. The high proportion of juvenile short rotation teak wood would have an influence on production, wood properties, and the end-product value. This drawback causes limited use of short rotation teak wood. Therefore, an environmentally friendly wood modification treatment is needed to improve the quality of short rotation teak wood.

Wood modification is means of a process that improves the properties of wood to produce a new material that disposed at the end of product life cycle without presenting environmental hazard any greater than unmodified wood [7]. Thermal modification has been the most important wood modification developed on industrial scale. In parallel to thermal modification, wood chemical modification constituted another attractive alternative to develop non biocide wood treatments. Even if a lot of

methods have been proposed at laboratory scale, only two of them have led to the development of industrial application, namely acetylation and furfurylation [8]. Wood modification with furfuryl alcohol (FA) is a non-toxic alternative method to replace the toxic conventional preservation treatments [9]. FA is derived from renewable resource (primarily corn cobs) and is improved by the polymerisation process. Research on furfurylated wood has been carried out for several decades. The furfurylation process has been developed since 1990s with the use of a catalyst that can increase the entry of FA into the wood. Sejati et al. [10] treated beech wood by furfurylation process using five different catalysts: maleic anhydride, maleic acid, citric acid, itaconic acid, and tartaric acid. It was noted in the study that tartaric acid can be considered as a new promising catalyst to perform furfurylated wood and good alternative to other catalysts since able to react with wood components allowing also fixation of FA polymer.

Furfurylation treatment leads to an improvement of several wood properties like dimension stability, modulus of elasticity (MOE), modulus of rupture (MOR), hardness, decay resistance [10,11,12]. Some studies have explained characteristics of furfurylated wood. Esteves et al. [11] treated wood with 70% furfuryl alcohol mixture and obtained anti-swelling efficiency (ASE) as high as 45% at a weight percent gain (WPG) of approximately 38%. Li et al. [13] reported a slight improvement of MOE and MOR. The furfurylation treatment has a much more significant effect on compressive strength than both MOE and MOR. Furfurylation treatment can improve durability against fungal decay [11,13,14], termites [15], marine borers [16], and weathering [17]. Recent studies regarding ecotoxicology and leachates from furfurylated wood have shown that furfurylated wood generally has a low ecotoxicity [9,14].

The present study was designed to investigate the effect of furfurylation treatment of short rotation teak to develop further industrial valorization on physical properties (WPG, density, and leachability), dimension stability (volumetric swelling, ASE, water uptake (WU), and bulking effect (BE)), mechanical properties (MOE and MOR), color change, decay resistance, and chemical compositions (holocellulose, hemicellulose,  $\alpha$ -cellulose, and lignin). Location of furfuryl alcohol in wood and in its different components was evaluated for the first time by Fourier Transform Infrared spectroscopy (FT-IR), Carbon 13 Nuclear Magnetic Resonance ( $^{13}\text{C}$  NMR), and Thermogravimetric Analysis (TGA).

## 2 Materials and Methods

### 2.1 Sample Preparation

The sample trees used were short rotation teak of 15 years with 25 cm diameter obtained from plantation forest managed by the state-owned enterprise (Perhutani) at Blora, Central Java (7°08'25.2"S/111°36'19.5"E) and short rotation teak of 8 years with diameter 25 cm obtained from SEAMEO BIOTROP at Bogor, West Java (6°38'07.7"S/106°49'32.8"E). Differences in growing conditions (environment, genetics, and silviculture) between Central Java and West Java resulted in variations in the teak growth. Blora, Central Java has an average rainfall below 1496-2506 mm/year and dry conditions for 4–6 months with an average temperature of 27 °C. Bogor, West Java, has a high annual rainfall (average 3500-4000 mm/year), and dry conditions for 2–3 months with an average temperature of 26 °C. From the felled tree, the log sections in length of 1 m were taken from each tree at the bottom part of the trees stem. The sample logs were sawn into 3 cm thickness of boards by bandsaw. Board samples were air dried up to a moisture

content of 12–15% and then sawn into smaller samples for studying various wood characteristics. Wood blocks for furfurylation treatment had a size of  $200 \times 20 \times 50 \text{ mm}^3$  (longitudinal x tangential x radial). Furfurylation was performed only on sapwood samples considering that heartwood presented sufficient natural durability and poor impregnability. For this purpose, smaller wood samples were taken from preceding wood blocks presenting only from sapwood.

## 2.2 Furfurylation Treatment

A mixture of FA with 97% in purity and tartaric acid as catalyst were used as the impregnation solution. Five percent (w/w) tartaric acid (based on the FA solution) was added to 45% furfuryl alcohol in aqueous solution. All samples before treatment were dried at the temperature  $103 \pm 2 \text{ }^\circ\text{C}$ , the weight ( $m_0$ ) and volume ( $V_0$ ) were recorded. The impregnation process was initiated with vacuum conditions 8–10 kPa for 5 min in an autoclave and followed by introduction of the aqueous FA solution. Afterward, the pressure was brought to 100 kPa (ambient pressure), then the wood samples were exposed to a pressure of 1200 kPa for 10 min. The impregnated samples were kept at room temperature for 48 h to evaporate the excess of water avoiding the appearance of cracks after polymerization. Samples were wrapped in aluminium foil to avoid furfuryl alcohol evaporation during curing and placed again in the reactor for polymerization. The reactor was closed and placed under nitrogen. The reactor temperature was slowly increased by  $0.5 \text{ }^\circ\text{C min}^{-1}$  from ambient to  $40 \text{ }^\circ\text{C}$  and maintained at this temperature for 10 h. After this period, the reactor temperature was increased by  $0.5 \text{ }^\circ\text{C min}^{-1}$  from 40 to  $120 \text{ }^\circ\text{C}$  and the temperature was maintained for 16 h. Heating was then stopped and furfurylated wood samples were allowed to cool down to room temperature under inert atmosphere. All treated woods were measured for their weight ( $m_1$ ) and volume ( $V_1$ ).

## 2.3 WPG, density, and BE

WPG was calculated using the following equation (Eq. 1):

$$\text{WPG (\%)} = ((m_1 - m_0) / m_0) \times 100 \quad (1)$$

where WPG is the percentage of weight gain due to the furfurylation treatment,  $m_0$  is the initial mass of wood at  $103 \text{ }^\circ\text{C}$  before treatment,  $m_1$  is the mass of wood after treatment.

Density was calculated from the following equation (Eq. 2):

$$\text{Density (gr cm}^{-3}\text{)} = m_0 / V_0 \text{ or } m_1 / V_1 \quad (2)$$

where  $m_0$  is the initial mass of wood at  $103 \text{ }^\circ\text{C}$  before treatment,  $m_1$  is the mass of wood after treatment,  $V_0$  is the initial volume of wood at  $103 \text{ }^\circ\text{C}$  before treatment,  $V_1$  is the volume of wood after treatment.

BE during wood treated was calculated from the following equation (Eq. 3):

$$\text{BE (\%)} = ((V_1 - V_0) / V_0) \times 100 \quad (3)$$

where BE is the percentage of bulk value of untreated or treated wood during treatment,  $V_0$  is the initial volume of wood at  $103 \text{ }^\circ\text{C}$  before treatment,  $V_1$  is the volume of wood after treatment.

## 2.4 Leaching test

The leaching test of untreated and furfurylated woods were carried out according to a procedure adapted from NF X 41-568 [18]. Sample section of  $30 \times 15 \times 5 \text{ mm}^3$  (L, R, T) was prepared in six replications for both untreated and furfurylated woods. The samples were submerged in test tubes of distilled water with a quantity of distilled water representing 5 times the volume of the introduced wood. The

leaching test for the samples were carried out in six leaching periods (1, 2, 4, 8, 16, 48 h) under continuous shaking (175 linear shakes  $\text{min}^{-1}$ ) at 20 °C. Between the periods of 4 and 8 h, used distilled water was removed and the wood samples were kept without water for 16 h in tube test. After leaching periods were completed, samples were dried at 103 °C for 48 h and reweighed ( $m_2$ ).

Weight loss due to leaching ( $W_{LL}$ ) was calculated from the following equation (Eq. 4):

$$W_{LL} (\%) = ((m_1 - m_2) / m_1) \times 100 \quad (4)$$

where  $W_{LL}$  is the percentage of weight loss value of untreated or furfurylated wood due to leaching,  $m_1$  is the mass of wood after treatment,  $m_2$  is the mass at 103 °C of untreated and treated wood after leaching process.

## 2.5 Chemicals compound analysis

Before the analysis, untreated and furfurylated wood samples were ground in Wiley mill to 40-60 mesh sawdust. The sawdust was extracted in Soxhlet apparatus for 24 h with different solvent of increasing polarity: dichloromethane, acetone, toluene/ethanol (2/1, v/v), and water. Two replicates of the extract free sawdust were measured for each wood in chemical composition. After determining chemical composition, holocellulose, cellulose, and lignin samples were then analysed with ATR-FTIR Perkin Elmer Spectrum 2000 instrument to indicate any reaction with the FA.

### 2.5.1 Holocellulose

The holocellulose was measured according to method developed by Rowell [19]. An 80 mL of hot distilled water was added to 2.5 g of wood sawdust, followed by 0.5 mL acetic acid, and 1 g of sodium chlorite in a 250 ml Erlenmeyer flask. The mixture was heated in a water bath at 70 °C. After 1 h, 0.5 mL of acetic acid and 1 g of sodium chlorite were added. Addition of 0.5 mL acetic acid and 1 g of sodium chlorite was repeated and it took 7 hours of chloriting. The sample could be left without further addition of acetic acid and sodium chlorite in the water bath for overnight. At the end of 24 hours of reaction, the sample was cooled and filtered on filter paper using a Buchner funnel until the yellow color and the odor of chlorine dioxide disappeared. The holocellulose was washed with acetone, vacuum oven dries at 105 °C for 24 hours, placed in a desiccator for an hour and weighed.

### 2.5.2 $\alpha$ -cellulose

The  $\alpha$ -cellulose was obtained by Rowel method [19]. A 2 g of oven dried holocellulose was placed into a 250 ml Erlenmeyer flask. Ten ml of 17.5% NaOH solution was added to the 250 ml Erlenmeyer flask and placed in water bath at 20°C. Further, 5 ml of 17.5% NaOH solution was added every 5 minutes during 15 minutes. The mixtures were conditioned at 20°C for 30 min. The mixture was then diluted with 33 ml distilled water and was kept for 1 hour at 20°C before filtering. All of the holocellulose residue were transferred to the crucible, and washed with 100 ml of 8.3% NaOH solution, 100 ml of distilled water, 15 ml of 10% acetic acid solution and finally 250 ml distilled water. The residue of  $\alpha$ -cellulose (white residue) was dried in an oven at 103 °C for 24 h, and weighed.

### 2.5.3 Hemicellulose

The hemicellulose content was calculated using this equation (Eq. 5):

$$\text{Hemicellulose (\%)} = \text{Holocellulose (\%)} - \alpha\text{-cellulose (\%)} \quad (5)$$

### 2.5.4 Lignin content

Klason lignin content was measured in this research work [20]. A 0.3 g of sawdust (extractive-free) and 3 ml of 72% v/v sulfuric acid was added to the tubes.



The tubes were placed into the water bath at 30 °C for 1 h. After 1 hour of reaction, samples were diluted by 84 ml with distilled water. The diluted samples were placed into the autoclaved for 1 h at 120°C and cooled at the room temperature. Finally, the lignin was obtained by Büchner filtration. Before rinsing off the solution with water, the soluble lignin was measured. The filtrate was diluted 20 times by distilled water, then its absorbance was measured at 205 nm by UV spectrometry. UV white was 3% sulfuric acid. Once the absorbance of the filtrate was achieved, the Klason lignin extraction could be continued with water rinses. The samples were finally dried overnight in an oven at 103 °C and weighed.

## 2.6 Fourier Transform Infrared (FT-IR) Spectroscopy

FT-IR spectrums were recorded on an ATR-FTIR Perkin Elmer Spectrum 2000 instrument. The untreated and furfurylated samples were in the range of 600 - 4000 cm<sup>-1</sup> at a resolution of 4 cm<sup>-1</sup> with an ATR cell.

## 2.7 Cross-Polarisation/Magic Angle Spinning (CP/MAS) <sup>13</sup>C NMR analysis

Solid state CP/MAS <sup>13</sup>C NMR spectra were recorded on a Bruker MSL300 spectrometer at a frequency of 75.47 MHz. Acquisition time was 0.026 s with number of transients of about 1200. All the spectra were run with a relaxation delay of 5 s, CP time of 1 ms and spectral width of 20,000 Hz. Spinning rates were 5 KHz. Interferograms were processed with Top Spin 3.6.2 using an apodization value of 21 Hz. Chemical shifts are expressed in parts per million (ppm).

## 2.8 Volumetric swelling, ASE, and WU measurement

The volumetric swelling, ASE, and WU measurement was measured by the method developed by Pfriem [21]. The test samples with a dimension of 10 × 20 × 20 mm<sup>3</sup> (L, R, T) were prepared in five replications from untreated and furfurylated wood. The untreated and furfurylated wood were dried at 103 °C and their weights (W<sub>d</sub>) and volumes were recorded (V<sub>d</sub>). Samples were directly immersed in distilled water and placed under vacuum condition (10 kPa) for 1 hour. After 24 h releasing the vacuum, the sample was released from the distillate water and their wet weight (W<sub>w</sub>) and volume (V<sub>w</sub>) were measured. Four cycles (1, 2, 3, and 14 days) of drying-soaking system were conducted and their weigh and volume (W<sub>d</sub>, V<sub>d</sub>, W<sub>w</sub>, and V<sub>w</sub>) for each cycle were recorded. The volumetric swelling (S<sub>v</sub>) and ASE were calculated. The volumetric swelling (S<sub>v</sub>) was determined using formula by equation (Eq. 6):

$$S_v (\%) = (V_w - V_d) / V_d \quad (6)$$

where S<sub>v</sub> is the percentage of volumetric swelling of wood sample, V<sub>w</sub> is the wet volumetric sample of the wood sample, and V<sub>d</sub> is the dry volumetric of the oven-dried sample at 103 °C.

ASE was calculated by equation (Eq. 7):

$$ASE (\%) = [(S_{vu} - S_{vt}) / S_{vu}] \times 100 \quad (7)$$

where ASE is the percentage of anti-swelling efficiency of treated wood, S<sub>vu</sub> is volumetric swelling of untreated wood, and S<sub>vt</sub> is the volumetric swelling of the treated wood.

WU was determined using the following equation (Eq. 8):

$$WU (\%) = [(W_w - W_d) / W_d] \times 100 \quad (8)$$

where WU is percentage of water uptake, W<sub>d</sub> is the weight of the air-dried sample at 103 °C before immersion and W<sub>w</sub> is the weight after immersion in water.

## 2.9 MOE and MOR

MOE and MOR of wood specimens were measured following the standard procedure of EN 310 [22] with the sample size of 200 x 20 x 5 mm<sup>3</sup> (L, R, T). Wood samples of both untreated and furfurylated were conditioned at the room with a temperature of at 22 ± 2 °C and relative humidity of 65 ± 5% RH, until constant mass. MOE and MOR were determined using at one-point bending by device INSTRON 4467 universal testing machine (Buckinghamshire, UK).

## 2.10 Color Changes

Color measurements were conducted on the CIE L\*, a\*, b\* system with X-Rite spectrophotometer. The test samples with a dimension of 70 × 50 × 20 mm<sup>3</sup> (L, R, T) were prepared in four replications from untreated and furfurylated wood. The color measurements were carried out on untreated and furfurylated wood samples. The L\*, a\*, and b\* parameters for each specimen were measured at six contiguous locations on the wood surface. Color changes ( $\Delta E$ ) were calculated based on CIELab in Eq. 9:

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (9)$$

where L\* is the lightness, a\* and b\* describes the chromatic coordinates on the green-red color, and blue yellow axes, respectively.  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$  are the differences between untreated and furfurylated wood and  $\Delta E$  is color changes.

## 2.11 Decay resistance

The decay resistance after leaching test was evaluated by following a method from EN 113 [23] standard. The wood samples consisted of untreated teak wood, furfurylated teak sapwood, and beech as a control wood. Dimension of samples were 30 x 10 x 5 mm<sup>3</sup> (L, R, T) with 8 replications for each treatment and their weights ( $W_0$ ) were measured. White rot fungi of *Coriolus versicolor* (CV) and brown rot fungus of *Pycnoporus sanguineus* (PS) and *Coniophora puteana* (CP) were inoculated on potato agar culture medium in petri dish and cultivated in an incubator at 22 ± 2°C temperature, 70 ± 5 % of relative humidity for 14 days. After mycelium permeated the culture disc, wood samples were put in each petri dish and then placed in incubator for 12 weeks in a climatic chamber (at 22 ± 2 °C and 70 ± 5% RH). Once the fungal exposure was completed, samples were cleaned carefully from the fungus. All samples were then oven dried at 103 °C for 48 h and weighed ( $W_1$ ). Durability class was determined according to EN 350-1 [24] standard usually used to classify wood species natural durability. The decay resistance was measured by weight loss (WL) according this equation (Eq. 10):

$$WL (\%) = ((W_0 - W_1) / W_0) \times 100 \quad (10)$$

where WL is percentage of weight loss, and  $W_0$  and  $W_1$  are dry weight of the samples before and after infection, respectively.

## 2.12 Thermogravimetric Analysis (TGA)

Thermal stability of untreated and furfurylated wood was carried out by TGA using a TGA/DSC1-TMA/SDTA 84Xe instrument from Mettler Toledo equipped with STARe v.11 fr System program. Sample powders (± 10 mg) were placed in alumina crucible. Thermogram were recorded using a heating rate of 10 °C /min from 25 to 700 °C under a nitrogen purge (20 ml.min<sup>-1</sup>).

## 3. Results and discussions

### 3.1 WPG, density, and leachability

The WPG, density, and leachability of untreated and furfurylated teak sapwoods are presented in Table 1. The average of WPG for furfurylated teak woods



varied between 19.48-20.94%, similar result was also reported by Hadi et al. [15]. These authors noted that accacia mangium wood, which possess similar density value ( $0.60 \text{ g cm}^{-3}$ ) with teak wood, presented WPG value around 20.1% after furfurylation treatment. Density value was correlated with the WPG, in which the increase in density of 10% caused the increase in the WPG of 20%. The polymerization of FA in the cell wall is important to impart new properties on the treated wood. Polymerization FA with tartaric acid affected WPG value after treatment [10]. Furfurylation treatment improves density because the cell walls are filled with polymerized FA [25]. Impregnation of wood with a polymer causes penetration of polymer and strengthen cell walls which occurs through the polymer filling the void space within the cell wall [7]. The result in Table 1 shows that the difference in leachability between untreated and furfurylated wood was observed to be 0.20 % for Plus Perhutani teak and 0.30% for Biotrop teak, which indicate a good fixation of poly-FA in the teak woods. Furfurylated Plus Perhutani teak presented relatively similar resistance to leaching compared to furfurylated Biotrop teak.

### 3.2 Chemical components

The effect of furfurylation treatment on chemical composition such as holocellulose,  $\alpha$ -cellulose, hemicellulose, and lignin are presented in Table 2. The result in Table 2 shows that the same tendency occurred on  $\alpha$ -celluloses, hemicellulose, and lignin contents between Plus Perhutani and Biotrop teak wood. Holocellulose content decreased from 65.22% to 58.99% for Plus Perhutani teak and from 64.28% to 56.51% for Biotrop teak.  $\alpha$ -cellulose content also decreased after furfurylation treatment from 41.91% to 32.47% for Plus Perhutani teak and from 43.68% to 31.33% for Biotrop teak. The decrease of holocellulose and  $\alpha$ -cellulose contents could be due to the introduction of an additional fraction of FA in the wood. The increase in lignin contents could be due to the interactions of FA polymer with lignin. The cross-linking reactions between the FA polymer and lignin are possible, although it has not been proven [7]. In this study, FTIR analysis was performed to prove the changes in native wood components due to furfurylation treatment.

### 3.3 FT-IR analysis

Figure 1 shows the FT-IR spectra of the untreated and furfurylated short rotation teak sapwoods over the 600 to 4000  $\text{cm}^{-1}$  wavenumber range. The FT-IR absorbance at around 3400  $\text{cm}^{-1}$  for the furfurylated teak wood, which assigned as the stretching vibration of the hydroxyl group in polysaccharides, showed a relative decrease in intensity compared to untreated teak wood. Furfurylated wood had a lower hydroxyl group intensity than untreated wood. The decrease in the intensity of the hydroxyl groups caused the wood more hydrophobic. The absorption band in the spectra between untreated and furfurylated wood were not significantly different especially in fingerprint area. There was no absorption band which indicates the presence of furan groups. This phenomenon indicates that the occurrence of a clear chemical interaction between FA and the wood should be proved. Native wood components from untreated and furfurylated wood were analyzed to determine potential reactions of FA with wood components after the furfurylation treatments. Holocellulose and  $\alpha$ -cellulose fractions presented no significant differences in spectra between the untreated and furfurylated samples. Another analysis was also performed to determine the effect of furfurylation treatment on native klason lignin.

Figure 2 shows the FT-IR spectra of all the Klason lignin fractions for the untreated and the furfurylated short rotation teak sapwoods over the 600 to 2000  $\text{cm}^{-1}$

<sup>1</sup> wavenumber range. The absorption bands of the furfurylated woods were different after furfurylation treatment. Two small bands were observed at 860 and 792 cm<sup>-1</sup>. These two small bands are assigned to aromatic C-H out-of-plane deformation and 2,5-disubstituted furan ring, respectively [12]. This result indicates that the polymerization of FA was completed and chemical interaction occurred between FA and lignin. Electrophilic aromatic substitutions involved in polymerization of FA were also very likely involved in reactions of FA or of its derivatives with lignin. These observations confirmed higher percentage of Klason lignin in furfurylated teak woods.

### 3.4 <sup>13</sup>C (CP-MAS) NMR analysis

The CP/MAS <sup>13</sup>C NMR spectra of the untreated and furfurylated short rotation teak sapwoods is presented in Figures 3. Both untreated wood samples showed classical signals identifiable to main wood polymer components. Cellulose's carbons appeared between 60 and 105 ppm. The signals comprised between 72 and 90 ppm were assigned to C2, C3, C4, C5 and C6 carbons, while the signal at 105 ppm was assigned to hemiacetalic C1 carbon. Two signals at 89 and 84 ppm were endorsed to the C4 carbon of crystalline and amorphous cellulose, respectively. Similar behaviour was observed for the signals at 65 and 62 ppm characteristic of the C6 carbon of crystalline and amorphous cellulose. Signals of hemicelluloses were less obvious due to their overlapping with signals of cellulose. The shoulder at 102 ppm on the C1 signal of cellulose was assigned to hemiacetal carbon of hemicelluloses, while carbons of acetyl groups were detected at 20 and 170 ppm. Methoxyl groups of syringyl and guaiacyl units were detected at 56 ppm, while aromatic carbons appeared between 110 and 155 ppm: aromatic carbons linked to hydrogen appeared around 110 ppm, those linked to carbon around 135 ppm and those linked to oxygen around 150 ppm. Aliphatic carbons of phenylpropane units of lignin were masked by signals of cellulose and hemicelluloses. No difference was detected between untreated Plus Perhutani and Biotrop woods. Furfurylated samples presented in both case, signals characteristic of FA polymer present in the wood structure. The signal at 28 ppm allocated to methylene bridges between aromatic structures including furan rings as well as benzenic rings. The increase of the signal at 155 ppm was characteristic the two alpha carbons of furan ring close to the oxygen atom indicating clearly presence of FA polymer. The two beta carbons of furan ring appeared at 110 and 120 ppm as a shoulder the C1 signal of cellulose. To confirm observations made by FT-IR analysis on the regioselectivity of the reaction of FA with the different wood cell wall polymers, lignin fractions of untreated and furfurylated Plus Perhutani and Biotrop woods were also analyzed by CP/MAS <sup>13</sup>C NMR (Figure 4). Spectra of furfurylated samples clearly showed characteristic signal of FA polymer in addition to classical lignin signals. The signal at 28 ppm was assigned to methylene bridges due to reaction of FA with itself or lignin. Increase of signals at 110 and 155 ppm were characteristic carbons of furan ring. The CP/MAS <sup>13</sup>C NMR spectra confirmed the presence of FA polymer either mixed with lignin polymer or copolymerized with lignin.

### 3.5 Volumetric swelling, ASE, WU, and BE

The volumetric swelling, ASE, WU, and BE of the untreated and furfurylated teak sapwoods are shown in Table 1. The volumetric swelling of furfurylated wood was smaller than that of untreated wood. The decreasing in volumetric swelling value was followed by the increasing in ASE value after furfurylation treatment. ASE of furfurylated Plus Perhutani and Biotrop teak wood reached 64.17% and 60.01%, respectively. Sejati et al. [10] reported similar ASE (approximately 66%) on

furfurylated beech wood containing 50% of FA and 5% of tartaric acid. Furfurylation treatment resulted in a decrease in WU values around 59% after furfurylation treatment. The previous result also reported the water uptake decrease after furfurylation treatment (around 50 %), which is due to the polymers filling the void spaces and blocking the water absorption [12]. It was reported in another study that higher ASE generates lower WU [26,27]. In addition, WU and ASE are improved due to the decrease in crystallinity. This may be attributed to the penetration of poly-FA into the amorphous region of the wood cell wall [26]. Furfurylation treatment can stabilize wood dimensions due to the reduction of void volume in wood [28]. The bulking agent will come in into the empty space in the cell wall to replace water, then the wood dimension becomes more stable [27,29]. BE values increased in the average of 11% after furfurylation treatment. The increase of the BE values suggested that adding the FA polymer by the impregnation process can improve dimensional stability. This result was also confirmed by FTIR analysis, in which furfurylated wood was difficult to absorb water due to decreasing in hydroxyl groups lead to the increase in dimensional stability.

### 3.6 MOE and MOR

MOE and MOR of the untreated and furfurylated short rotation teak sapwood are presented in Figure 5. The mean MOE values for untreated Plus Perhutani and untreated Biotrop were 13703.13 kg cm<sup>-2</sup> and 13941.98 kg cm<sup>-2</sup>, respectively. The MOE value of furfurylated Plus Perhutani was almost similar as the untreated, meanwhile MOE of furfurylated Biotrop decreased by 14%. This result was in line with a report by Pfriem et al. [21], who found reduction in MOE in furfurylated beech wood catalyzed by maleic anhydride. Another study also reported that furfurylation treatment did not significantly change the MOE values [11,13,30]. The MOR value decreased with furfurylation treatment by 20% from 128.12 kg cm<sup>-2</sup> to 102.58 kg cm<sup>-2</sup> for Plus Perhutani teak wood and by 37% from 153.90 kg cm<sup>-2</sup> to 97.06 kg cm<sup>-2</sup> for Biotrop teak wood. This result could be due to the decrease in the cellulose content.

### 3.7 Color changes

Table 3 presents the variation of  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ , and  $\Delta E^*$  of untreated and furfurylated short rotation teak sapwood. The color of untreated wood tended to change after furfurylation treatment. The change of  $L^*$  value indicated the change in brightness in which the color of the furfurylated teak wood was darker. The furfurylated woods presented smaller  $L^*$  value for both short rotation teak woods when compared to the untreated wood. The decrease in  $L^*$  value of furfurylated pine wood was also reported by Acosta et al. [31]. Furfurylated short rotation teak woods resulted in the increase in  $a^*$  value, while the  $b^*$  value was not changed after furfurylation treatment. Dong et al. [25] reported that the  $a^*$  value of furfurylated wood increases compared with untreated and  $b^*$  value varies among species. Generally,  $a^*$  value has correlation to the phenolic compound on the wood [32]. Colors between untreated and furfurylated wood were prominently different. Mean value of total color difference ( $\Delta E$ ) changed dramatically after furfurylation treatment. The darker color is generated by the conjugated structure which causes the natural color of wood is covered by the color of poly-FA [33].

### 3.8 Decay resistance

The weight losses of untreated and furfurylated short rotation teak sapwood against the white rot fungi and brown rot fungi are presented in Figure 6. Durability of

the furfurylated wood against fungus increased after furfurylation treatment. Weight losses of Plus Perhutani teak wood due to PS, CV, and CP decreased after furfurylation treatment from 15.53% to 1.02%; from 29.63% to 2.92%; and from 1.87% to 1.06%, respectively. Weight losses of Biotrop teak wood due to PS, CV, and CP decreased after furfurylation treatment from 12.67% to 1.06%; from 19.76% to 1.23%; and from 1.63% to 1.16%, respectively. Beech wood controls presented the highest weight losses confirming the fungus tested in this work destroyed hardly the wood. Other studies also reported a decrease in weight loss of pine wood due to CP [11,34] and CV [13]. According to EN 350-1, furfurylated short rotation teak wood can be categorized in durability class 1 (very durable). Furfurylation treatment results in physical blocking of the entrance of decay and the reduction of equilibrium moisture content due to the penetration of resin into the wood cell wall and cross-linkage between FA and wood cell wall components [35]. It contributes to inhibiting fungus growth in furfurylated wood to get the moisture required for decay [36].

### 3.9 TGA analysis

Thermal stability of untreated and furfurylated short rotation teak wood were investigated by the thermogravimetric analysis. Figure 7 shows the TGA and differential thermogravimetric (DTG) curves of the untreated and furfurylated short rotation teak sapwood. The details of major decomposition temperatures and the corresponding percentage of weight losses are summarized in Table 4. The first degradation region from 50 to 100 °C is related to the evaporation of moisture and adsorbed water from the wood. The weight losses of the furfurylated wood were lower than untreated wood at this temperature range (Figure 7). The second degradation occurs from 220 to 300 °C the weight loss decreases abruptly, while from 300 °C to above the weight loss decreases gradually. The final degradation occurs around 450 °C and corresponds to degradation of  $\alpha$ -cellulose and lignin, meanwhile the carbonisation of lignin and furfuryl alcohol polymer were extended up to 650 °C [37,38]. DTG curve is attributed to lignin decomposition at a slower rate than hemicellulose and cellulose [39]. Compared with the untreated, the furfurylated wood showed a higher  $T_{onset}$  and  $T_{max}$  (Table 4). For furfurylated woods, the  $T_{onset}$  of degradation (temperature at 5% weight loss) were 221 °C for Plus Perhutani teak wood and 220 °C for Biotrop teak wood, which are higher than untreated wood. Meanwhile,  $T_{max}$  degradation temperatures were found at 356 °C in furfurylated Plus Perhutani and at 359 °C in furfurylated Biotrop. The weight losses for all the samples were about 70-80% at the end of the measurement (700 °C).  $T_{onset}$  and  $T_{max}$  increased after furfurylation treatment. Furfurylated samples presented higher thermal stability due to higher stability of remaining carbonized lignin and furfuryl alcohol polymer. This result confirms with Acosta et al. [40] who reported that furfurylated juvenile pine wood with higher lignin is more thermally stable than furfurylated mature pine wood.

### 4. Conclusion

Furfurylation allows to improve properties of short rotation teak sapwood. Dimensional stability of the furfurylated wood is increased, though MOE and MOR are decreased. Furfurylation treatment causes the darker color on the short rotation teak sapwood. Durability against decaying fungi of the furfurylated short rotation teak wood is classified as very durable. The furfurylation treatment causes changes in the  $\alpha$ -cellulose, hemicellulose, and lignin contents of the short rotation teak sapwood, the latter one increasing, while the two first ones decrease. FT-IR and NMR analysis



indicate the presence of chemical bonds between FA or FA-polymer and lignin. TGA and DTG analyses indicated that the furfurylated teak wood presents better thermal stability, compared to the untreated teak wood. The furfurylation treatment can be eco-friendly and valuable method for improving the quality of short rotation teaks for high quality wood product utilization.

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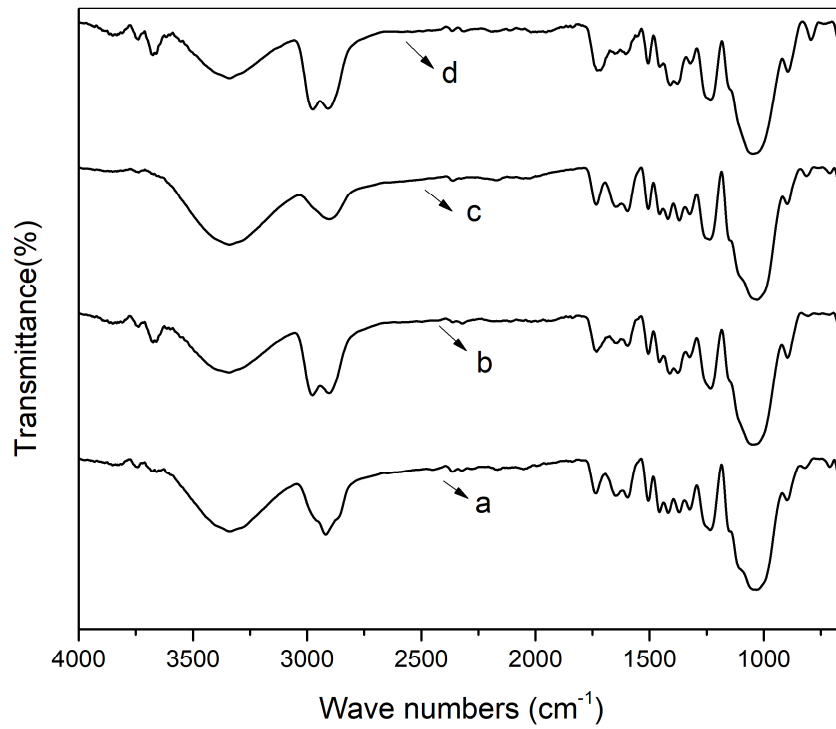
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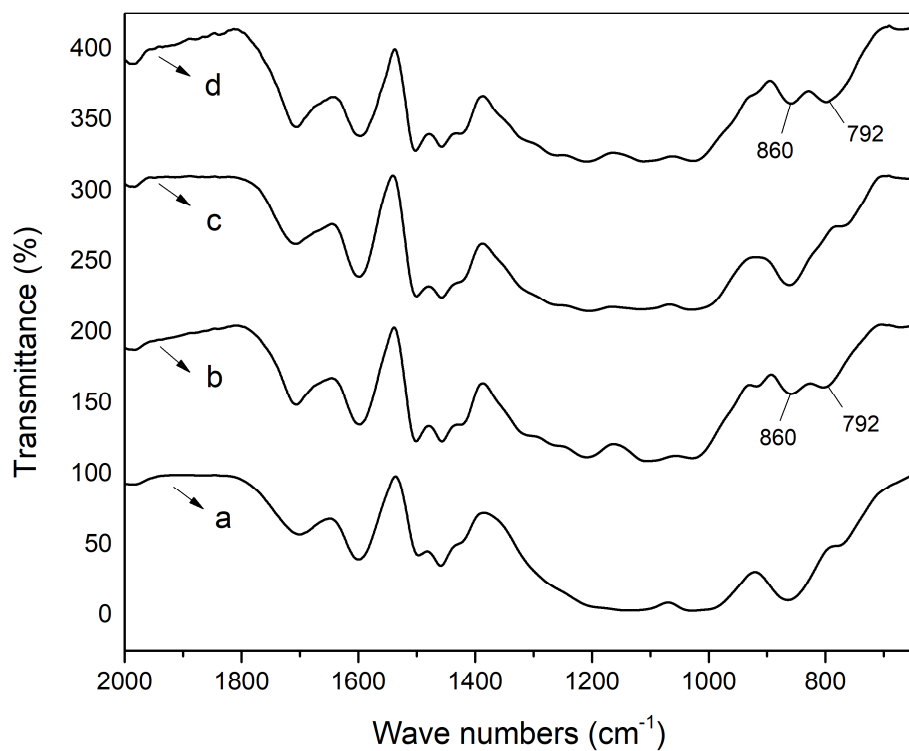


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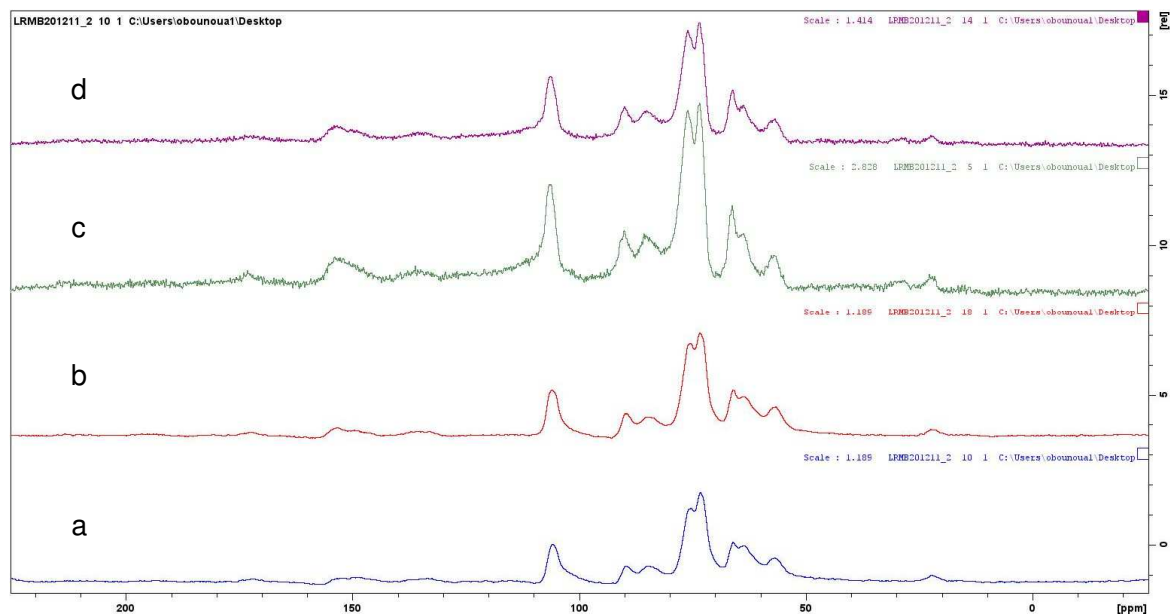
**Figure 1.** FT-IR spectra of untreated Plus Perhutani teak wood (a); and furfurylated Plus Perhutani teak wood (b); untreated Biotrop teak wood (c); and furfurylated Biotrop teak wood (d)



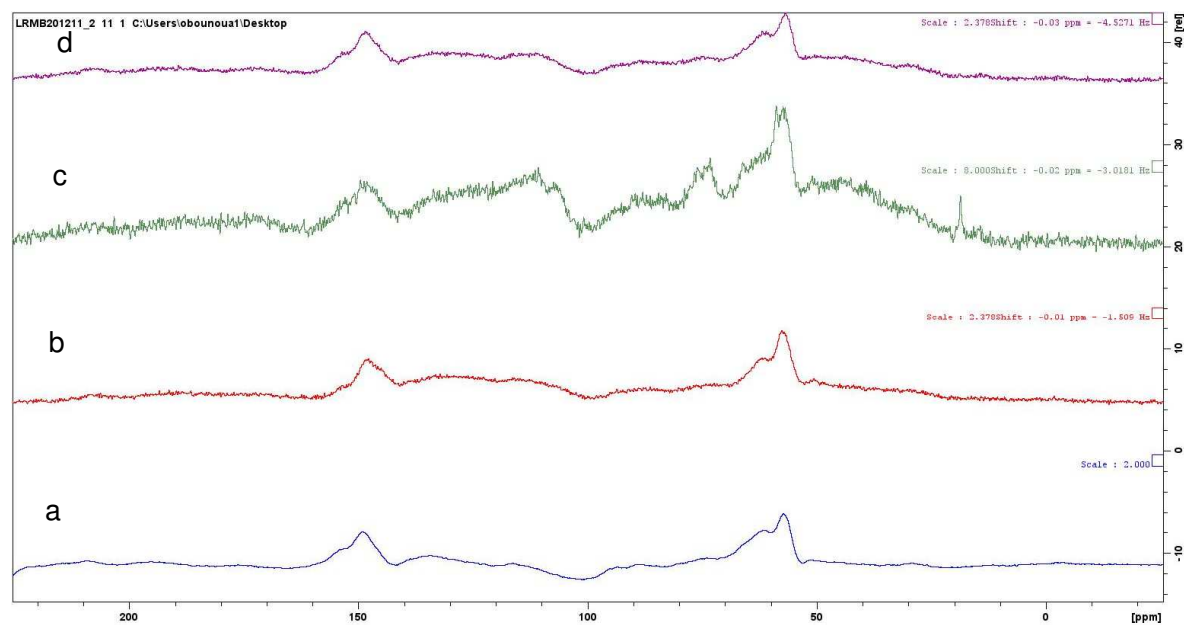
**Figure 2.** FT-IR spectra of native klason lignin of untreated Plus Perhutani teak wood (a); and furfurylated Plus Perhutani teak wood (b); untreated Biotrop teak wood (c); and furfurylated Biotrop teak wood (d)



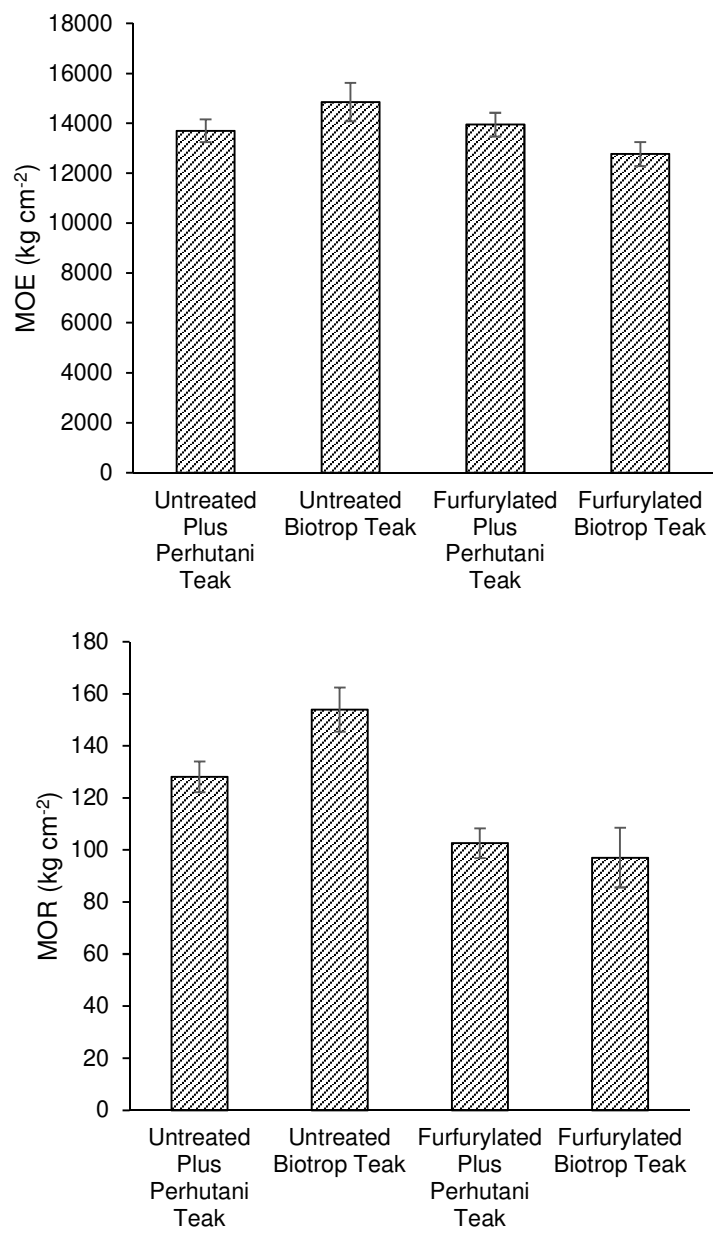
**Figure 3.** CP/MAS  $^{13}\text{C}$  NMR spectra of untreated Plus Perhutani teak wood (a); untreated Biotrop teak wood (b); furfurylated Plus Perhutani teak wood (c) and furfurylated Biotrop teak wood (d)



**Figure 4.** CP/MAS  $^{13}\text{C}$  NMR spectra of untreated Plus Perhutani teak lignin (a); untreated Biotrop teak lignin (b); furfurylated Plus Perhutani teak lignin (c) and furfurylated Biotrop teak lignin (d)

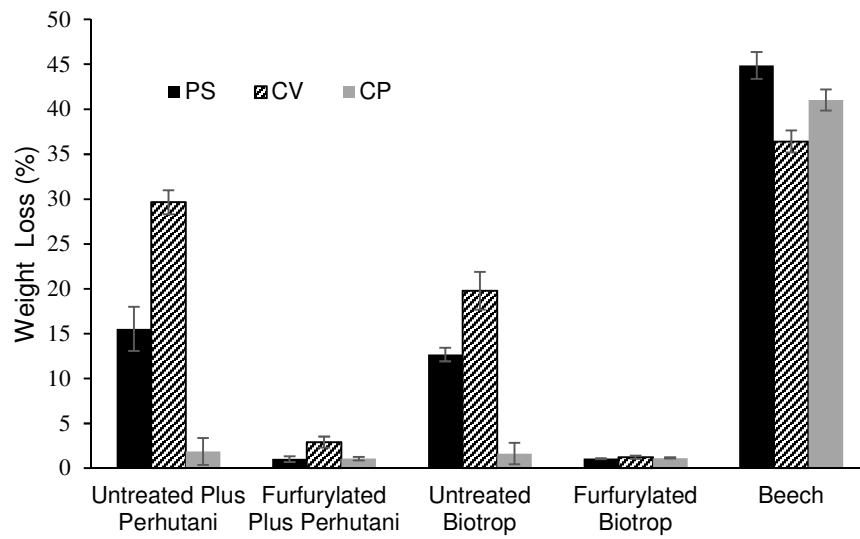


**Figure 5.** MOE and MOR of untreated and furfurylated of short rotation teak sapwoods



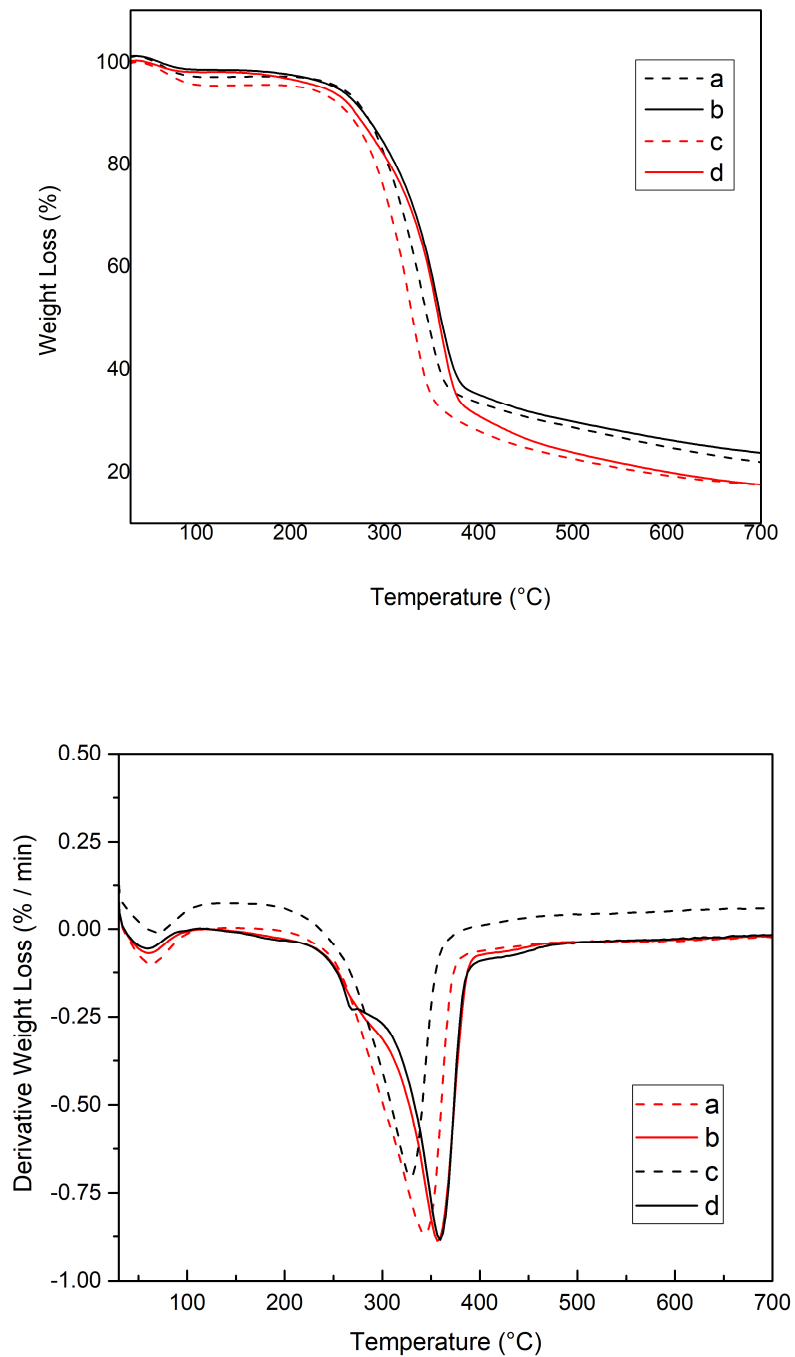


**Figure 6.** Weight losses of short rotation teak sapwoods to decay fungus



Note : PS = *Pycnoporus sanguineus* ; CV = *Coriolus versicolor* ; CP = *Coniophora puteana*

**Figure 7.** TGA and DTG curve of untreated Plus Perhutani teak wood (a); furfurylated Plus Perhutani teak wood (b); untreated Biotrop teak wood (c); and furfurylated Biotrop teak wood (d)



**Table 1.** WPG, density, leachability, and dimensional stability of untreated and furfurylated short rotation teak sapwoods

Parameters	Untreated Plus Perhutani	Untreated Biotrop	Furfurylated Plus Perhutani	Furfurylated Biotrop
WPG (%)	0	0	20.94±2.76	19.48±1.60
Density (g cm <sup>-3</sup> )	0.52±0.01	0.58±0.01	0.61±0.01	0.64±0.02
WL <sub>L</sub> (%)	1.96±0.08	1.44±0.20	2.17±0.34	1.75±0.12
Volumetric swelling (%)	11.74±0.59	8.86±0.68	5.64±0.50	5.12±0.62
ASE (%)	0	0	64.17±3.16	60.01±4.82
Water uptake (%)	94.67±1.14	92.71±2.96	37.14±4.28	31.23±4.33
BE (%)	0	0	12.48±0.73	10.56±1.61

**Table 2.** Chemical composition of untreated and furfurylated of short rotation teak woods

Chemical composition	Untreated Plus Perhutani	Untreated Biotrop	Furfurylated Plus Perhutani	Furfurylated Biotrop
Holocellulose (%)	65.22±3.48	64.28±0.01	58.99±1.65	56.51±0.27
$\alpha$ -cellulose (%)	41.91±3.29	43.68±0.01	32.47±2.21	31.33±0.05
Hemicellulose (%)	23.31±0.20	20.60±0.04	26.52±0.56	25.18±0.32
Lignin (%)	36.37±0.45	31.60±1.15	37.31±0.06	38.84±0.67

**Table 3.** Color parameters for the untreated and furfurylated teak sapwoods

	Untreated Plus Perhutani	Furfurylated Plus Perhutani	Untreated Biotrop	Furfurylated Biotrop
L*	76.17±2.74	41.69±2.24	74.30±1.48	43.14±1.78
a*	5.28±0.63	10.42 ±0.58	5.37±0.79	10.39 ±0.67
b*	20.06±1.80	20.25±1.08	23.26±1.17	21.03±1.08
Δ E	-	34.95±3.94	-	31.70±2.41

**Table 4.** TGA results of untreated and furfurylated teak sapwoods

Species	Decomposition temperature		Weight loss (%) <sup>a</sup>	Residue (%) <sup>b</sup>
	T <sub>onset</sub> (°C)	T <sub>max</sub> (°C)		
Untreated Plus Perhutani	209.94	342.33	52.83	14.59
Furfurylated Plus Perhutani	221.32	356.50	53.08	16.90
Untreated Biotrop	215.40	328.17	57.52	21.17
Furfurylated Biotrop	220.99	359.33	54.31	20.20

<sup>a</sup> at the wood decomposition temperature<sup>b</sup> at 700 °C