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

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18

1 **Abstract**

2 Short rotation teak wood has low quality especially in dimensional stability and
3 durability. Furfurylation treatment was applied to improve technological properties of
4 the short rotation teak wood. The purpose of the study was to improve dimensional
5 stability and durability through furfurylation treatment. Short rotation teak sapwoods
6 (Plus Perhutani and Biotrop teaks) were treated through the impregnation process of
7 45% of furfuryl alcohol (FA) and 5% of tartaric acid as catalyst followed by heated in
8 oven at 120 °C for 16 h under nitrogen atmosphere. Dimensional stability was
9 characterized by volumetric swelling, anti-swelling efficiency (ASE), water uptake
10 (WU), and bulking effect (BE), meanwhile decay durability was determined by the
11 weight loss (WL). The results show that ASE increased in the average of 64%, WU
12 decreased in the average of 59%, and BE increased in the average of 11%, which
13 indicated the improvement of dimensional stability. Weight losses of furfurylated teak
14 woods due to fungal decay of *Coriolus versicolor* (CV), *Pycnoporus sanguineus* (PS),
15 and *Coniophora puteana* (CP) were in the average of 2%, 1%, and 1%, respectively,
16 which categorized their durability to be class 1 (very durable). The increase of lignin
17 content showed by Klason lignin content measurement and Fourier Transform
18 Infrared (FTIR) and Carbon 13 Nuclear Magnetic Resonance (¹³C NMR) analysis
19 indicated that the reaction of FA with lignin occurred after furfurylation treatment.
20 Furfurylated sapwood presented also better thermic stability compared to untreated
21 wood by Thermogravimetric Analysis (TGA). The FA treatment can be applied for
22 improving the quality of short rotation teaks for high quality wood product utilization.

23
24 Keywords: dimensional stability, decay durability, FTIR and TGA analysis, furfuryl
25 alcohol, lignin content, short rotation teak

26 27 **1. Introduction**

28 The supply of long rotation teak wood (*Tectona grandis* Linn. F.) has been
29 declining year after year. The amount of long rotation teak wood production in
30 Indonesia in 2017, the first quarter was 134500 m³ and decreased in the fourth
31 quarter to 115841 m³ [1]. Short rotation teak is one of the solutions to overcome the
32 limited supply of long rotation teak wood. Short rotation teak is known by various
33 trade name, such as Biotrop teak is the short rotation teak planted by SEAMEO
34 Biotrop and Plus Perhutani Teak is developed by the Perhutani Research and
35 Development Center. Short rotation teak has several advantages such as short
36 cutting cycle (7-15 years), lesser branches, straight and cylindrical trunk. However,
37 short rotation teak wood has low strength, density and durability, and high proportion
38 of juvenile wood [2,3]. Juvenile wood has shorter fibres with thinner walls, larger
39 microfibril angles, lower densities, and lower strength properties compared to mature
40 wood [4,5,6]. The high proportion of juvenile short rotation teak wood would have an
41 influence on production, wood properties, and the end-product value. This drawback
42 causes limited use of short rotation teak wood. Therefore, an environmentally friendly
43 wood modification treatment is needed to improve the quality of short rotation teak
44 wood.

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

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

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

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

35 **2 Materials and Methods**

36 **2.1 Sample Preparation**

37 The sample trees used were short rotation teak of 15 years with 25 cm
38 diameter obtained from plantation forest managed by the state-owned enterprise
39 (Perhutani) at Blora, Central Java (7°08'25.2"S/111°36'19.5"E) and short rotation
40 teak of 8 years with diameter 25 cm obtained from SEAMEO BIOTROP at Bogor,
41 West Java (6°38'07.7"S/106°49'32.8"E). Differences in growing conditions
42 (environment, genetics, and silviculture) between Central Java and West Java
43 resulted in variations in the teak growth. Blora, Central Java has an average rainfall
44 below 1496-2506 mm/year and dry conditions for 4–6 months with an average
45 temperature of 27 °C. Bogor, West Java, has a high annual rainfall (average 3500-
46 4000 mm/year), and dry conditions for 2–3 months with an average temperature of
47 26 °C. From the felled tree, the log sections in length of 1 m were taken from each
48 tree at the bottom part of the trees stem. The sample logs were sawn into 3 cm
49 thickness of boards by bandsaw. Board samples were air dried up to a moisture
50

1 content of 12–15% and then sawn into smaller samples for studying various wood
 2 characteristics. Wood blocks for furfurylation treatment had a size of **200 x 20 x 50**
 3 **mm³** (longitudinal x tangential x radial). Furfurylation was performed only on sapwood
 4 samples considering that heartwood presented sufficient natural durability and poor
 5 impregnability. For this purpose, smaller wood samples were taken from preceding
 6 wood blocks presenting only from sapwood.

8 **2.2 Furfurylation Treatment**

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

28 **2.3 WPG, density, and BE**

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

$$30 \text{ WPG (\%)} = ((m_1 - m_0) / m_0) \times 100 \quad (1)$$

31 where WPG is the percentage of weight gain due to the furfurylation treatment, m_0 is
 32 the initial mass of wood at 103 °C before treatment, m_1 is the mass of wood after
 33 treatment.

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

$$35 \text{ Density (gr cm}^{-3}\text{)} = m_0 / V_0 \text{ or } m_1 / V_1 \quad (2)$$

36 where m_0 is the initial mass of wood at 103 °C before treatment, m_1 is the mass of
 37 wood after treatment, V_0 is the initial volume of wood at 103 °C before treatment, V_1
 38 is the volume of wood after treatment.

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

$$40 \text{ BE (\%)} = ((V_1 - V_0) / V_0) \times 100 \quad (3)$$

41 where BE is the percentage of bulk value of untreated or treated wood during
 42 treatment, V_0 is the initial volume of wood at 103 °C before treatment, V_1 is the
 43 volume of wood after treatment.

45 **2.4 Leaching test**

46 The leaching test of untreated and furfurylated woods were carried out
 47 according to a procedure adapted from NF X 41-568 [18]. Sample section of 30×15
 48 $\times 5$ mm³ (L, R, T) was prepared in six replications for both untreated and furfurylated
 49 woods. The samples were submerged in test tubes of distilled water with a quantity
 50 of distilled water representing 5 times the volume of the introduced wood. The

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

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

$$8 \quad W_{LL} (\%) = ((m_1 - m_2) / m_1) \times 100 \quad (4)$$

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

12 **2.5 Chemicals compound analysis**

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

21 **2.5.1 Holocellulose**

22 The holocellulose was measured according to method developed by Rowell
23 [19]. An 80 mL of hot distilled water was added to 2.5 g of wood sawdust, followed by
24 0.5 mL acetic acid, and 1 g of sodium chlorite in a 250 ml Erlenmeyer flask. The
25 mixture was heated in a water bath at 70 °C. After 1 h, 0.5 mL of acetic acid and 1 g
26 of sodium chlorite were added. Addition of 0.5 mL acetic acid and 1 g of sodium
27 chlorite was repeated and it took 7 hours of chloriting. The sample could be left
28 without further addition of acetic acid and sodium chlorite in the water bath for
29 overnight. At the end of 24 hours of reaction, the sample was cooled and filtered on
30 filter paper using a Buchner funnel until the yellow color and the odor of chlorine
31 dioxide disappeared. The holocellulose was washed with acetone, vacuum oven
32 dries at 105 °C for 24 hours, placed in a desiccator for an hour and weighed.

33 **2.5.2 α -cellulose**

34 The α -cellulose was obtained by Rowel method [19]. A 2 g of oven dried
35 holocellulose was placed into a 250 ml Erlenmeyer flask. Ten ml of 17.5% NaOH
36 solution was added to the 250 ml Erlenmeyer flask and placed in water bath at 20°C.
37 Further, 5 ml of 17.5% NaOH solution was added every 5 minutes during 15 minutes.
38 The mixtures were conditioned at 20°C for 30 min. The mixture was then diluted with
39 33 ml distilled water and was kept for 1 hour at 20°C before filtering. All of the
40 holocellulose residue were transferred to the crucible, and washed with 100 ml of
41 8.3% NaOH solution, 100 ml of distilled water, 15 ml of 10% acetic acid solution and
42 finally 250 ml distilled water. The residue of α -cellulose (white residue) was dried in
43 an oven at 103 °C for 24 h, and weighed.

44 **2.5.3 Hemicellulose**

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

$$46 \quad \text{Hemicellulose (\%)} = \text{Holocellulose (\%)} - \alpha\text{-cellulose (\%)} \quad (5)$$

47 **2.5.4 Lignin content**

48 Klason lignin content was measured in this research work [20]. A 0.3 g of
49 sawdust (extractive-free) and 3 ml of 72% v/v sulfuric acid was added to the tubes.
50

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

11 2.6 Fourier Transform Infrared (FT-IR) Spectroscopy

12 FT-IR spectrums were recorded on an ATR-FTIR Perkin Elmer Spectrum
 13 2000 instrument. The untreated and furfurylated samples were in the range of 600 -
 14 4000 cm⁻¹ at a resolution of 4 cm⁻¹ with an ATR cell.

16 2.7 Cross-Polarisation/Magic Angle Spinning (CP/MAS) ¹³C NMR analysis

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

24 2.8 Volumetric swelling, ASE, and WU measurement

25 The volumetric swelling, ASE, and WU measurement was measured by the
 26 method developed by Pfriem [21]. The test samples with a dimension of 10 × 20 × 20
 27 mm³ (L, R, T) were prepared in five replications from untreated and furfurylated
 28 wood. The untreated and furfurylated wood were dried at 103 °C and their weights
 29 (W_d) and volumes were recorded (V_d). Samples were directly immersed in distilled
 30 water and placed under vacuum condition (10 kPa) for 1 hour. After 24 h releasing
 31 the vacuum, the sample was released from the distillate water and their wet weight
 32 (W_w) and volume (V_w) were measured. Four cycles (1, 2, 3, and 14 days) of drying-
 33 soaking system were conducted and their weigh and volume (W_d, V_d, W_w, and V_w) for
 34 each cycle were recorded. The volumetric swelling (S_v) and ASE were calculated.
 35 The volumetric swelling (S_v) was determined using formula by equation (Eq. 6):

$$36 S_v (\%) = (V_w - V_d) / V_d \quad (6)$$

37 where S_v is the percentage of volumetric swelling of wood sample, V_w is the wet
 38 volumetric sample of the wood sample, and V_d is the dry volumetric of the oven-dried
 39 sample at 103 °C.

40 ASE was calculated by equation (Eq. 7):

$$41 ASE (\%) = [(S_{vu} - S_{vt}) / S_{vu}] \times 100 \quad (7)$$

42 where ASE is the percentage of anti-swelling efficiency of treated wood, S_{vu} is
 43 volumetric swelling of untreated wood, and S_{vt} is the volumetric swelling of the
 44 treated wood.

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

$$46 WU (\%) = [(W_w - W_d) / W_d] \times 100 \quad (8)$$

47 where WU is percentage of water uptake, W_d is the weight of the air-dried sample at
 48 103 °C before immersion and W_w is the weight after immersion in water.

50 2.9 MOE and MOR

1 MOE and MOR of wood specimens were measured following the standard
 2 procedure of EN 310 [22] with the sample size of 200 x 20 x 5 mm³ (L, R, T). Wood
 3 samples of both untreated and furfurylated were conditioned at the room with a
 4 temperature of at 22 ± 2 °C and relative humidity of 65 ± 5% RH, until constant mass.
 5 MOE and MOR were determined using at one-point bending by device INSTRON
 6 4467 universal testing machine (Buckinghamshire, UK).

8 2.10 Color Changes

9 Color measurements were conducted on the CIE L*, a*, b* system with X-Rite
 10 spectrophotometer. The test samples with a dimension of 70 × 50 × 20 mm³ (L, R, T)
 11 were prepared in four replications from untreated and furfurylated wood. The color
 12 measurements were carried out on untreated and furfurylated wood samples. The L*,
 13 a*, and b* parameters for each specimen were measured at six contiguous locations
 14 on the wood surface. Color changes (ΔE) were calculated based on CIELab in Eq. 9:

$$15 \quad \Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (9)$$

16 where L* is the lightness, a* and b* describes the chromatic coordinates on the
 17 green-red color, and blue yellow axes, respectively. ΔL^* , Δa^* , Δb^* are the
 18 differences between untreated and furfurylated wood and ΔE is color changes.

20 2.11 Decay resistance

21 The decay resistance after leaching test was evaluated by following a method
 22 from EN 113 [23] standard. The wood samples consisted of untreated teak wood,
 23 furfurylated teak sapwood, and beech as a control wood. Dimension of samples were
 24 30 x 10 x 5 mm³ (L, R, T) with 8 replications for each treatment and their weights
 25 (W_0) were measured. White rot fungi of *Coriolus versicolor* (CV) and brown rot
 26 fungus of *Pycnoporus sanguineus* (PS) and *Coniophora puteana* (CP) were
 27 inoculated on potato agar culture medium in petri dish and cultivated in an incubator
 28 at 22 ± 2°C temperature, 70 ± 5 % of relative humidity for 14 days. After mycelium
 29 permeated the culture disc, wood samples were put in each petri dish and then
 30 placed in incubator for 12 weeks in a climatic chamber (at 22 ± 2 °C and 70 ± 5%
 31 RH). Once the fungal exposure was completed, samples were cleaned carefully from
 32 the fungus. All samples were then oven dried at 103 °C for 48 h and weighed (W_1).
 33 Durability class was determined according to EN 350-1 [24] standard usually used to
 34 classify wood species natural durability. The decay resistance was measured by
 35 weight loss (WL) according this equation (Eq. 10):

$$36 \quad WL (\%) = ((W_0 - W_1) / W_0) \times 100 \quad (10)$$

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

40 2.12 Thermogravimetric Analysis (TGA)

41 Thermal stability of untreated and furfurylated wood was carried out by TGA
 42 using a TGA/DSC1-TMA/SDTA 84Xe instrument from Mettler Toledo equipped with
 43 STARe v.11 fr System program. Sample powders (± 10 mg) were placed in alumina
 44 crucible. Thermogram were recorded using a heating rate of 10 °C /min from 25 to
 45 700 °C under a nitrogen purge (20 ml.min⁻¹).

47 3. Results and discussions

48 3.1 WPG, density, and leachability

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

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

17 3.2 Chemical components

18 The effect of furfurylation treatment on chemical composition such as
19 holocellulose, α -cellulose, hemicellulose, and lignin are presented in Table 2. The
20 result in Table 2 shows that the same tendency occurred on α -celluloses,
21 hemicellulose, and lignin contents between Plus Perhutani and Biotrop teak wood.
22 Holocellulose content decreased from 65.22% to 58.99% for Plus Perhutani teak and
23 from 64.28% to 56.51% for Biotrop teak. α -cellulose content also decreased after
24 furfurylation treatment from 41.91% to 32.47% for Plus Perhutani teak and from
25 43.68% to 31.33% for Biotrop teak. The decrease of holocellulose and α -cellulose
26 contents could be due to the introduction of an additional fraction of FA in the wood.
27 The increase in lignin contents could be due to the interactions of FA polymer with
28 lignin. The cross-linking reactions between the FA polymer and lignin are possible,
29 although it has not been proven [7]. In this study, FTIR analysis was performed to
30 prove the changes in native wood components due to furfurylation treatment.

32 3.3 FT-IR analysis

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

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

¹ wavenumber range. The absorption bands of the furfurylated woods were different after furfurylation treatment. Two small bands were observed at 860 and 792 cm⁻¹. 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 ¹³C (CP-MAS) NMR analysis

The CP/MAS ¹³C 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 ¹³C 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 ¹³C 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

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

17 18 **3.6 MOE and MOR**

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

30 31 **3.7 Color changes**

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

47 48 **3.8 Decay resistance**

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

1 the furfurylated wood against fungus increased after furfurylation treatment. Weight
2 losses of Plus Perhutani teak wood due to PS, CV, and CP decreased after
3 furfurylation treatment from 15.53% to 1.02%; from 29.63% to 2.92%; and from
4 1.87% to 1.06%, respectively. Weight losses of Biotrop teak wood due to PS, CV,
5 and CP decreased after furfurylation treatment from 12.67% to 1.06%; from 19.76%
6 to 1.23%; and from 1.63% to 1.16%, respectively. Beech wood controls presented
7 the highest weight losses confirming the fungus tested in this work destroyed hardly
8 the wood. Other studies also reported a decrease in weight loss of pine wood due to
9 CP [11,34] and CV [13]. According to EN 350-1, furfurylated short rotation teak wood
10 can be categorized in durability class 1 (very durable). Furfurylation treatment results
11 in physical blocking of the entrance of decay and the reduction of equilibrium
12 moisture content due to the penetration of resin into the wood cell wall and cross-
13 linkage between FA and wood cell wall components [35]. It contributes to inhibiting
14 fungus growth in furfurylated wood to get the moisture required for decay [36].
15

16 3.9 TGA analysis

17 Thermal stability of untreated and furfurylated short rotation teak wood were
18 investigated by the thermogravimetric analysis. Figure 7 shows the TGA and
19 differential thermogravimetric (DTG) curves of the untreated and furfurylated short
20 rotation teak sapwood. The details of major decomposition temperatures and the
21 corresponding percentage of weight losses are summarized in Table 4. The first
22 degradation region from 50 to 100 °C is related to the evaporation of moisture and
23 adsorbed water from the wood. The weight losses of the furfurylated wood were
24 lower than untreated wood at this temperature range (Figure 7). The second
25 degradation occurs from 220 to 300 °C the weight loss decreases abruptly, while
26 from 300 °C to above the weight loss decreases gradually. The final degradation
27 occurs around 450 °C and corresponds to degradation of α -cellulose and lignin,
28 meanwhile the carbonisation of lignin and furfuryl alcohol polymer were extended up
29 to 650 °C [37,38]. DTG curve is attributed to lignin decomposition at a slower rate
30 than hemicellulose and cellulose [39]. Compared with the untreated, the furfurylated
31 wood showed a higher T_{onset} and T_{max} (Table 4). For furfurylated woods, the T_{onset} of
32 degradation (temperature at 5% weight loss) were 221 °C for Plus Perhutani teak
33 wood and 220 °C for Biotrop teak wood, which are higher than untreated wood.
34 Meanwhile, T_{max} degradation temperatures were found at 356 °C in furfurylated Plus
35 Perhutani and at 359 °C in furfurylated Biotrop. The weight losses for all the samples
36 were about 70-80% at the end of the measurement (700 °C). T_{onset} and T_{max}
37 increased after furfurylation treatment. Furfurylated samples presented higher
38 thermal stability due to higher stability of remaining carbonized lignin and furfuryl
39 alcohol polymer. This result confirms with Acosta et al. [40] who reported that
40 furfurylated juvenile pine wood with higher lignin is more thermally stable than
41 furfurylated mature pine wood.
42

43 4. Conclusion

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

1 indicate the presence of chemical bonds between FA or FA-polymer and lignin. TGA
2 and DTG analyses indicated that the furfurylated teak wood presents better thermal
3 stability, compared to the untreated teak wood. The furfurylation treatment can be
4 eco-friendly and valuable method for improving the quality of short rotation teaks for
5 high quality wood product utilization.
6
7

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18

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44

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)

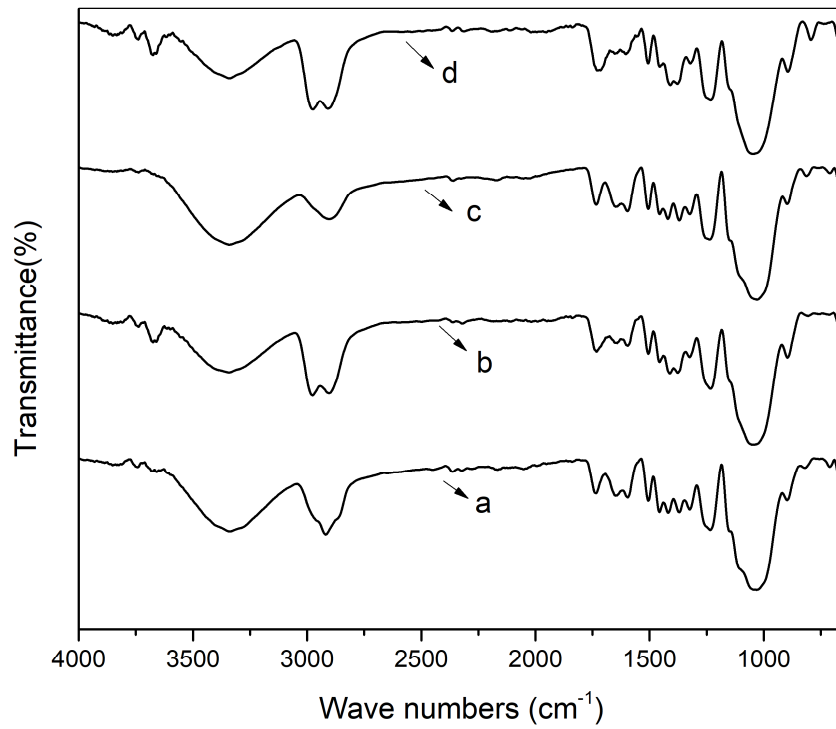


Figure 2. FT-IR spectra of native klason lignin of untreated Plus Perhutani teak wood (a); and furfurylated Plus Perhutani teak wood (b); untreated Biotrop teak wood (c); and furfurylated Biotrop teak wood (d)

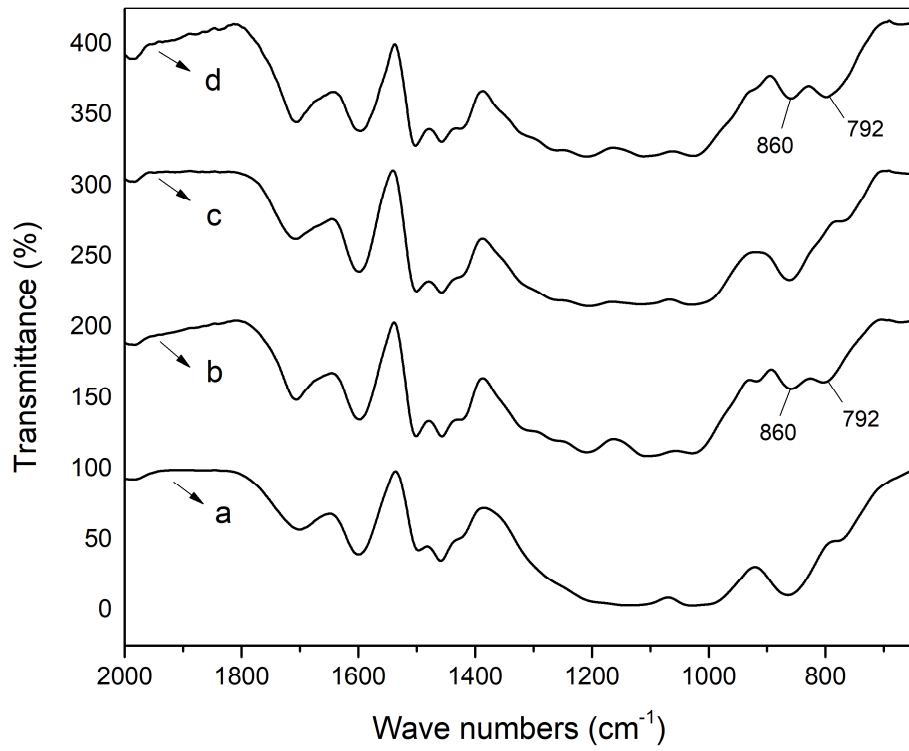


Figure 3. CP/MAS ^{13}C NMR spectra of untreated Plus Perhutani teak wood (a); untreated Biotrop teak wood (b); furfurylated Plus Perhutani teak wood (c) and furfurylated Biotrop teak wood (d)

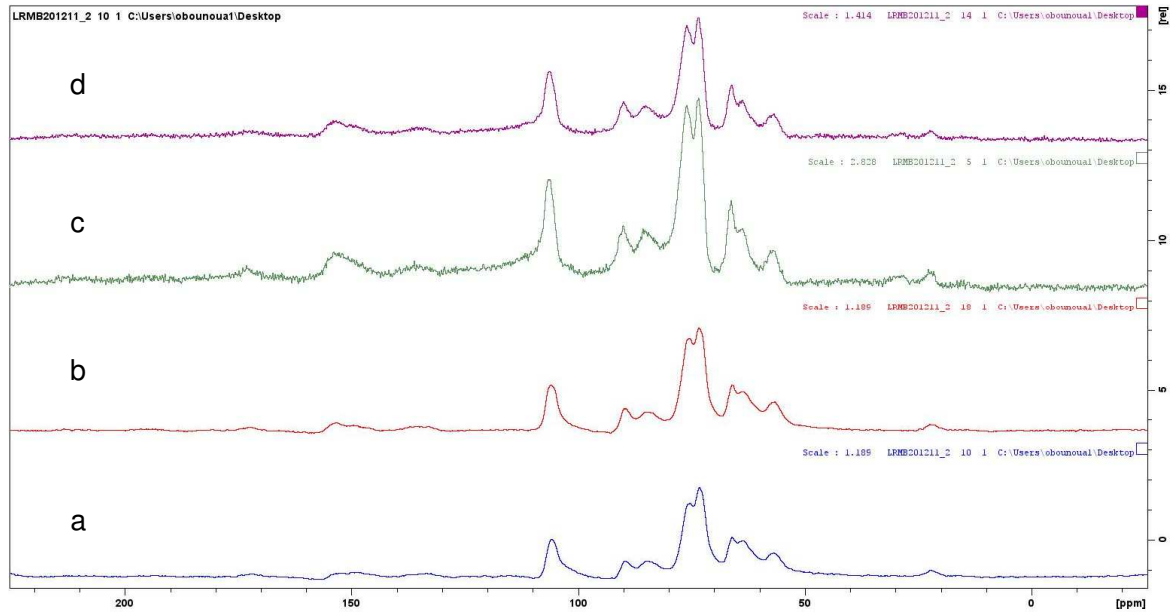


Figure 4. CP/MAS ^{13}C NMR spectra of untreated Plus Perhutani teak lignin (a); untreated Biotrop teak lignin (b); furfurylated Plus Perhutani teak lignin (c) and furfurylated Biotrop teak lignin (d)

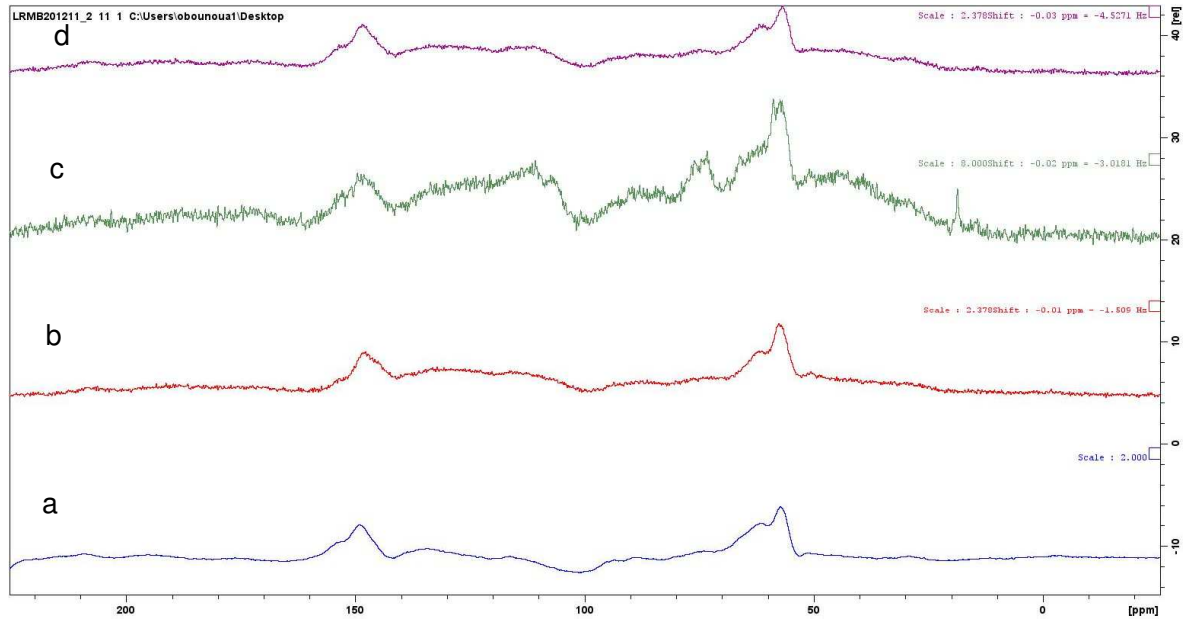


Figure 5. MOE and MOR of untreated and furfurylated of short rotation teak sapwoods

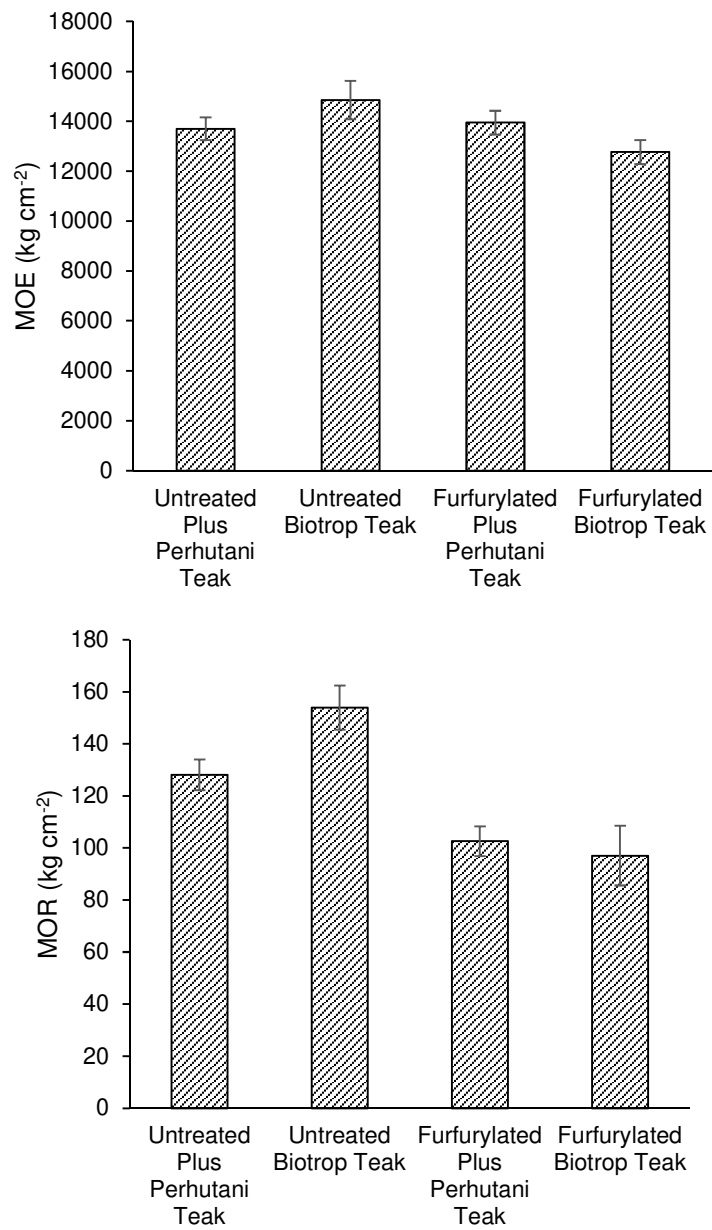
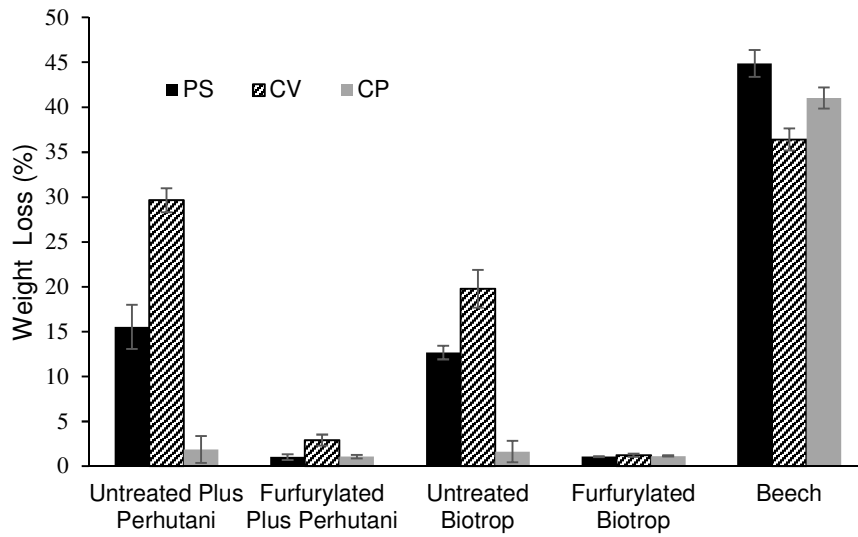


Figure 6. Weight losses of short rotation teak sapwoods to decay fungus



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)

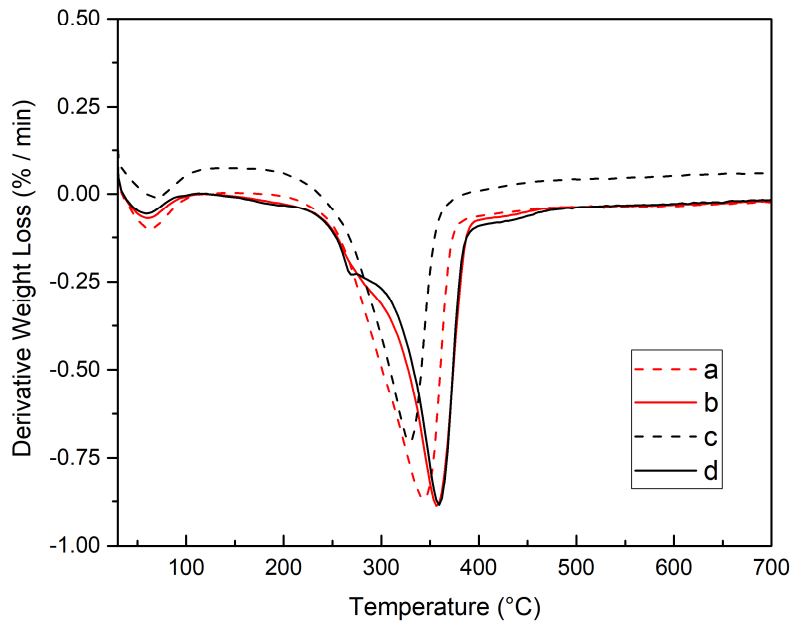
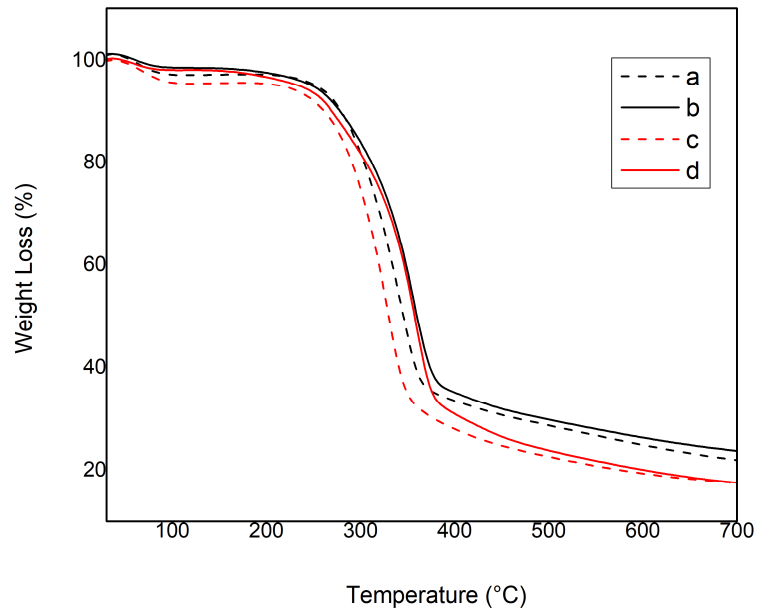


Table 1. WPG, density, leachability, and dimensional stability of untreated and furfurylated short rotation teak sapwoods

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 ⁻³)	0.52±0.01	0.58±0.01	0.61±0.01	0.64±0.02
WL _L (%)	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
α -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 (%) ^a	Residue (%) ^b
	T _{onset} (°C)	T _{max} (°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

^a at the wood decomposition temperature^b at 700 °C