

## Article

# Effect of Citric Acid and Benzophenone Tetracarboxylic Acid Treatments on Stability, Durability, and Surface Characteristic of Short Rotation Teak

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**Abstract:** Short rotation teak (SRT) has a diameter of around 30 cm at the age of 15 years; however, the wood still has low quality, especially in dimensional stability and biological durability. The objective of this study was to improve dimensional stability and biological durability of SRT through modification treatment using non-biocide chemicals, citric acid (CA) and benzophenone tetracarboxylic acid (BPTCA). SRT sapwood was impregnated by 20% and 40% of CA or BPTCA followed by heating in the oven at 150 °C for 2 h. Dimensional stability, strength, surface characteristics and durability against termite attacks of the modified woods were evaluated. Results show that dimensional stability as anti-swelling efficiency (ASE) increased by an average of 46% (CA) and 50% (BPTCA) after treatments. Durability against termites showed that the modified woods were categorized to be class 1 (very durable). Fourier Transform Infrared (FTIR) and X-ray Diffraction observations indicated a possible chemical reaction between the wood components with CA and BPTCA. The water-based acrylic and oil-based alkyd contact angle values of the modified woods were lower than 90°, indicating their high wetting ability against the varnish liquids. The treatment can be applied to improve the quality of SRT for high-quality wood product utilization.

**Keywords:** dimensional stability; non-biocide treatment; surface characteristic; strength; termite resistance



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## 1. Introduction

In Indonesia, high-quality teak wood (*Tectona grandis* Linn. F.) from long rotation (60 to 80 years) plantations has been declining year after year. The total production of long-rotation teak in 2018 was 1,304,909 m<sup>3</sup>, but this value then decreased by 27% to be 953,372 m<sup>3</sup> in 2019 [1]. Hence, short rotation teak/SRT (10–15 years) plantation should be a promising solution to overcome the scarcity supply of teak wood from long rotation. SRT plantation has been increasing, in which the total area managed by state-owned forests (PERHUTANI) was around 300 Ha in 2021. Recently, SRT plantation has also been developed by other communities and private companies. SRT plantations produce tree diameters of 25 to 30 cm. These trees have fewer branches with straight and cylindrical trunks. However, SRT wood has low density, stability, and durability, and a high proportion of juvenile wood [2,3]. Juvenile wood has shorter fibers with thinner walls, and larger microfibril angles, which lead to lower strength compared to mature wood [4,5]. The presence of a high juvenile portion in the SRT wood could affect its processing during the production of high-value products. Therefore, a promising green wood modification treatment is needed to improve the quality and finally to increase the utilization of the SRT wood for high-quality wood products.

The wood modification was proposed to improve the quality of wood [6]. Thermal modification has been the most important wood modification developed on an industrial scale. An innovative eco-friendly thermal modification was investigated to improve the quality of SRT [7]. The results indicated that the anti-swelling efficiency (ASE) value after heat treatment was improved, ranging between 12.9% and 46.7%, indicating an improvement in dimensional stability. It was also reported in the study that the surface roughness and SFE values decrease as the heating temperature increases. The teak wood treated at 220 °C provides the K-values (0.04 for acrylic and 5.04 for alkyd paint) larger than 0, which indicates good wettability. However, heat treatment at 220 °C decreases the MOE and MOR values of the SRT and does not provide any improvement in durability against termites [8]. It was reported in another study that heat treatment at 220 °C increases the durability of the SRT against decay fungi (*Trametes versicolor*) from moderate to very durable [8].

In parallel to thermal modification, chemical modification constitutes another attractive alternative as a promising wood treatment method. Even if a lot of methods have been proposed at the laboratory scale for chemical modification, only two of them have led to the development of industrial application, namely acetylation and furfurylation [9]. Due to the non-toxic property of these chemically modified woods, these techniques became an alternative to the non-biocidal method to replace some toxic conventional wood preservation treatments. Esteves et al. [10] treated wood with a 70% furfuryl alcohol mixture and obtained anti-swelling efficiency (ASE) as high as 45% at a weight percent gain (WPG) of approximately 38%. Furfurylation treatment leads to an improvement of several wood properties such as dimension stability, modulus of elasticity (MOE), modulus of rupture (MOR), hardness, and decay resistance [10–13]. Furfurylation treatment can improve durability against fungal decay [10,13], termites [14], and marine borers [6]. Recent studies regarding ecotoxicology and leachates of furfurylated wood have indicated that the furfurylated wood has a low ecotoxicity [15].

Furfurylation treatment was already applied to improve the technological properties of short-rotation teak wood [16]. This study reported that the short rotation teak impregnated with furfuryl alcohol (FA) 45% and catalyst 5%, followed by the heating process at 120 °C, presents excellent results in dimensional stability and decay durability. The study also disclosed that there is a reaction between FA and the wood lignin, presenting higher Klason lignin content isolated from the modified wood. From this study, further investigation on the use of other environmentally friendly chemicals to improve the low quality of short rotation teak is of interest and needs to be scrutinized.

Chemical modification of SRT using non-biocide glycerol-maleic anhydride (GMA) followed by heat treatment at 150 and 220 °C has also been investigated. The treatments could improve the technological properties of SRT wood, especially dimensional stability (increased 62%), decay (*Coriolus versicolor*, *Pycnoporus sanguineus*, and *Coniophora puteana*), and termite resistances [17]. Although both treatments presents excellent biological durability against decay and subterranean termites in the field, the mechanical properties of treatment at 220 °C decreases severely compared to 150 °C. In addition, since the wood, especially that used for exterior uses, undergoes harsh conditions due to weather changes, any degradation of wood, particularly lignin due to UV light, should be minimized [18]. Therefore, to improve durability and service life for this exterior use, wood modification and surface coating could become alternative methods. Surface characteristics (e.g., surface roughness and wettability) are a critical factor in surface coating.

The results presented above give an indication that the decrease in the strength of the modified woods could be caused by the high temperature (larger than 150 °C) imposed during the modification processes. Innovation in environmentally friendly non-biocide chemicals which could be cured at lower temperatures needs to be proposed. The present study was designed to investigate the effect of non-biocidal CA and BPTCA treatment of SRT to develop further industrial valorization on physical properties (weight percent gain (WPG), density, and leachability), dimension stability (anti-swelling efficiency (ASE), water

uptake (WU)), bending strength (MOE and MOR), termite resistance (weight loss, and defective samples), surface characteristics (roughness and wettability). The interaction of CA and BPTCA with the wood components was evaluated by Fourier Transform Infrared spectroscopy (FTIR) and X-ray diffraction instruments.

## 2. Materials and Methods

### 2.1. Sample Preparation

The sample trees of SRT of 20 years with around 40 cm diameter were obtained from plantation forests managed by the state-owned enterprise (Perhutani) at Blora, Central Java (7°08′25.2″ S/111°36′19.5″ E). Blora, Central Java, has an average rainfall between 1496 and 2506 mm/year, and experiences dry conditions for 4–6 months per year with an average temperature of 27 °C. Log sections with a length of 1 m were taken from each tree sample at the bottom part of the stem. The logs were transported to the workshop and band-sawed into boards in thickness of 3 cm. Board samples were air-dried up to a moisture content of 12%–15% and then sawed into smaller samples for modification tests and characterizations. Test samples for CA and BPTCA treatment were prepared in sizes of 200 × 20 × 50 mm (longitudinal × tangential × radial) with five replicates. The impregnation of CA and BPTCA was performed on sapwood samples, considering that heartwood presented sufficient natural durability and poor impregnability.

### 2.2. Citric Acid and Benzophenone Tetracarboxylic Acid Treatment

Both CA and BPTCA were ordered in the form of powder and used as the impregnation solution. Both CA and BPTCA of 20% and 40% concentration each were prepared in an aqueous solution. All of the test samples were dried at a temperature of  $103 \pm 2$  °C before treatment, and the weight ( $m_0$ ) and volume ( $V_0$ ) were measured. The impregnation process was initiated with vacuum conditions 8–10 kPa for 5 min in an autoclave and followed by the introduction of the aqueous CA and BPTCA solution. Afterward, the test samples in the CA and BPTCA solutions 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 water. The impregnated samples were wrapped in aluminum foil to avoid CA and BPTCA evaporation during curing and placed again in an oven for polymerization. The oven temperature was slowly increased by  $0.5$  °C  $\text{min}^{-1}$  from ambient to 100 °C and maintained at this temperature for 1 h. After this period, the oven temperature was increased by  $0.5$  °C  $\text{min}^{-1}$  to 150 °C (HT150 °C) and was maintained for 2 h. Heating was then stopped and treated wood samples were allowed to cool down to room temperature under an inert atmosphere. All treated woods were measured for their weight ( $m_1$ ) and volume ( $V_1$ ).

### 2.3. Weight Percent Gain (WPG) and Density Measurement

WPG was calculated using the following equation (Equation (1)):

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

where WPG is the percentage of weight gain due to the Citric Acid and Benzophenone Tetra Cyclic Acid treatment (five replicates for each treatment),  $m_0$  is the initial mass of wood at 103 °C before treatment,  $m_1$  is the mass of wood after treatment.

Density ( $n = 5$ ) was calculated from the following equation (Equation (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 °C before treatment,  $m_1$  is the mass of wood after treatment,  $V_0$  is the initial volume of wood at 103 °C before treatment,  $V_1$  is the volume of wood after treatment.

#### 2.4. Leaching Test

The leaching tests of untreated and treated SRT woods were carried out according to a procedure adapted from NF X 41-568 [19]. Test samples of  $30 \times 15 \times 5$  mm (L, R, T) were prepared in six replications for both untreated and treated woods. The leaching test samples were submerged in test tubes containing distilled water, with a quantity of distilled water representing 5 times the volume of the introduced samples. The leaching test for the samples was carried out in a leaching period of 24 h at 25 °C. Between the periods of 12 h, the used distilled water was removed and the wood samples were kept without water for 16 h in a 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 (WLL) was calculated from the following equation (Equation (3)):

$$\text{WLL (\%)} = ((m_1 - m_2)/m_1) \times 100 \quad (3)$$

where WLL is the percentage of weight loss of untreated or treated 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 the leaching process.

#### 2.5. Anti-Swelling Efficiency (ASE) and Water Uptake (WU) Measurement

The ASE and WU were measured by the method developed by Pfriem et al. [20]. Dimensional stability test samples in dimensions of  $10 \times 20 \times 20$  mm (L, R, T) were prepared in five replications from untreated and treated wood. The untreated and treated wood were dried at 103 °C and their weights ( $W_d$ ) and volumes were recorded ( $V_d$ ). The samples were directly immersed in distilled water and placed under vacuum conditions (10 kPa) for 1 h. After 24 h imposed in the vacuum, the samples were released from the distillate water and their green weight ( $W_w$ ) and volume ( $V_w$ ) were measured. Four cycles (1, 2, 3, and 14 days) of the drying-soaking system were conducted and their weight and volume ( $W_d$ ,  $V_d$ ,  $W_w$ , and  $V_w$ ) for each cycle were recorded. The volumetric swelling ( $S_v$ ) and ASE were calculated. The volumetric swelling ( $S_v$ ) was determined using the formula below (Equation (4)):

$$S_v (\%) = (V_w - V_d)/V_d \quad (4)$$

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

ASE was calculated by equation (Equation (5)):

$$\text{ASE (\%)} = [(S_{vu} - S_{vt})/S_{vu}] \times 100 \quad (5)$$

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

Water uptake (WU) ( $n = 5$ ) was determined using the following equation (Equation (6)):

$$\text{WU (\%)} = [(W_w - W_d)/W_d] \times 100 \quad (6)$$

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

#### 2.6. Fourier Transform Infrared (FTIR) Spectroscopy and X-ray Diffractometer (XRD)

FTIR spectrums were recorded on an ATR-FTIR Perkins Elmer Spectrum 2000 instrument in the range of  $600\text{--}4000\text{ cm}^{-1}$  at a resolution of  $4\text{ cm}^{-1}$  with an ATR cell both for untreated and treated samples.

The degree of crystallinity of the modified teak specimens was analyzed by means of an X-ray diffraction instrument (XRD 7000 Shimadzu), with the  $\theta$  range of  $10\text{--}60$ , the scan speed of  $1\text{ degree/min}$ , the X-ray tube of Cu (copper).

### 2.7. Modulus of Elasticity (MOE) and Modulus of Rupture (MOR)

MOE and MOR were measured following the standard procedure of EN 310 [21] with a sample size of  $200 \times 20 \times 5$  mm (L, R, T). Wood samples ( $n = 5$ ) of both untreated and treated samples were conditioned at the room with a temperature of  $22 \pm 2$  °C and a relative humidity of  $65\% \pm 5\%$  RH, until constant mass. MOE and MOR of static bending were determined by the INSTRON 4467 universal testing machine (Buckinghamshire, UK).

### 2.8. Field Test for Termite Resistance

The untreated and treated stakes ( $200 \times 20 \times 10$  mm (L, R, T);  $n = 5$ ) were oven-dried at 60 °C until constant weight (W0). All stakes were embedded in-ground to determine their resistance against subterranean termites at the research field of the Faculty of Forestry, IPB University, Indonesia. The stakes were buried vertically 150 mm below ground level and placed randomly at a distance of 300 mm among each other. The tests began between mid-December 2021 and mid-March 2022 (a period of 3 months). The stakes were removed after 12 weeks, washed with water, cleaned with a brush, and dried under sunlight, followed by drying in the oven at 60 °C until constant weight (W1). The stake's mass loss and rating due to the termite attack were calculated. The tested stakes were evaluated according to SNI 7207 [22] (Table 1). The mass loss (ML) was measured according to Equation (7):

$$ML (\%) = ((W0 - W1)/W0) \times 100 \quad (7)$$

where ML is the percentage of mass loss, and W0 and W1 are the dry weights of the samples before and after field exposure, respectively.

**Table 1.** The grading system based on weight loss of stakes by attacked subterranean termites [22].

| Durability Class | Resistance      | Mass Loss (%) |
|------------------|-----------------|---------------|
| I                | Very resistance | <3.52         |
| II               | Resistance      | 3.52–7.50     |
| III              | Moderate        | 7.50–10.96    |
| IV               | Poor            | 10.96–18.94   |
| V                | Very poor       | >18.94        |

The tested stakes were also investigated according to ASTM D 1758 [23] (Table 2). The wood damage grading system in Table 2 is classified based on the percentage of damages of cross-section due to attack by subterranean termites.

**Table 2.** The grading system based on percentage of damages stakes attacked by subterranean termite [23].

| Visual Rating | Description of Damage                                 |
|---------------|-------------------------------------------------------|
| 10            | Sound timber, no termite attack                       |
| 9             | Trace of termite attack less than 3% of cross section |
| 8             | Termites attack 3% to 10% of cross-section            |
| 7             | Termites attack 10% to 30% cross-section              |
| 6             | Termites attack 30% to 50% cross-section              |
| 4             | Termites attack 50% to 75% cross-section              |
| 0             | Failure due to termites attack                        |

### 2.9. Surface Roughness and Wettability Measurement

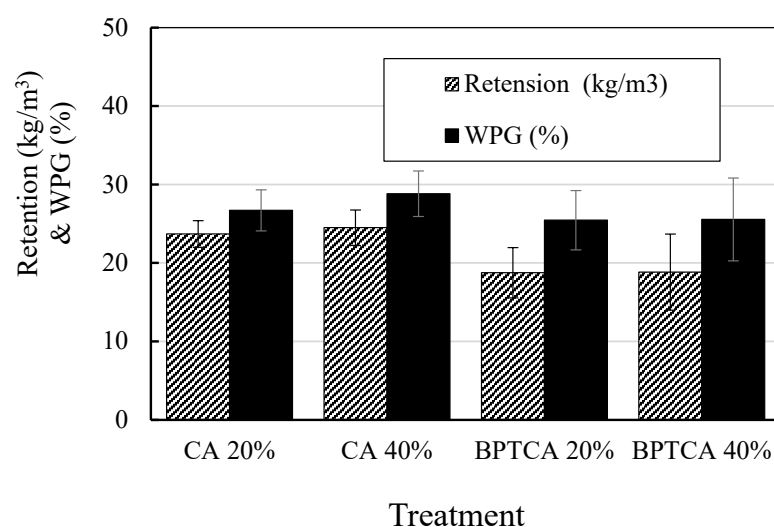
The measurement of surface roughness of wood specimens was performed perpendicularly to the fiber direction at five different positions on the tangential surface of each sample (five replicates) using a Mitutoyo type SJ-210 tester. The measurement according to ISO 4287 [24] was performed with a diamond tip radius of 5 µm, tracing the length of 6 mm, a cut-off of 0.8 mm, and a speed of 0.5 mm/s. The variable evaluated was the arithmetical mean roughness (Ra) value. The dynamic contact angles of alkyd and acrylic varnishes

for measurement of wettability were performed with a video measuring system with a high-resolution CCD camera. During measurement, the teak wood specimen (untreated and CA BA treated) was placed on the top of a table in front of the CCD video camera. The drop of selected standard liquids and the acrylic paint with a volume of 20  $\mu\text{L}$  was dropped by a syringe with a screwing method to obtain the same droplets. The drop shapes on the wood surface were captured by the CCD camera and saved for a duration of 180 s. Five droplets per sample were captured for each standard liquid and acrylic paint for the measurements of contact angle. Each of the captured video images was cut to an individual image at intervals of 10 s for total duration of 180 s. The Image-J 1.46 software with drop-snakes plugin analysis was used to measure the contact angle ( $\theta$ ) of the individual image of the drop. The contact angles of each droplet on the surface of the wood specimen were measured both on the left side and the right side of the droplet, and then the values were averaged. The contact angle tests were conducted in a room with a temperature of  $23 \pm 2$  °C and a relative humidity of  $80\% \pm 5\%$ .

### 3. Results & Discussions

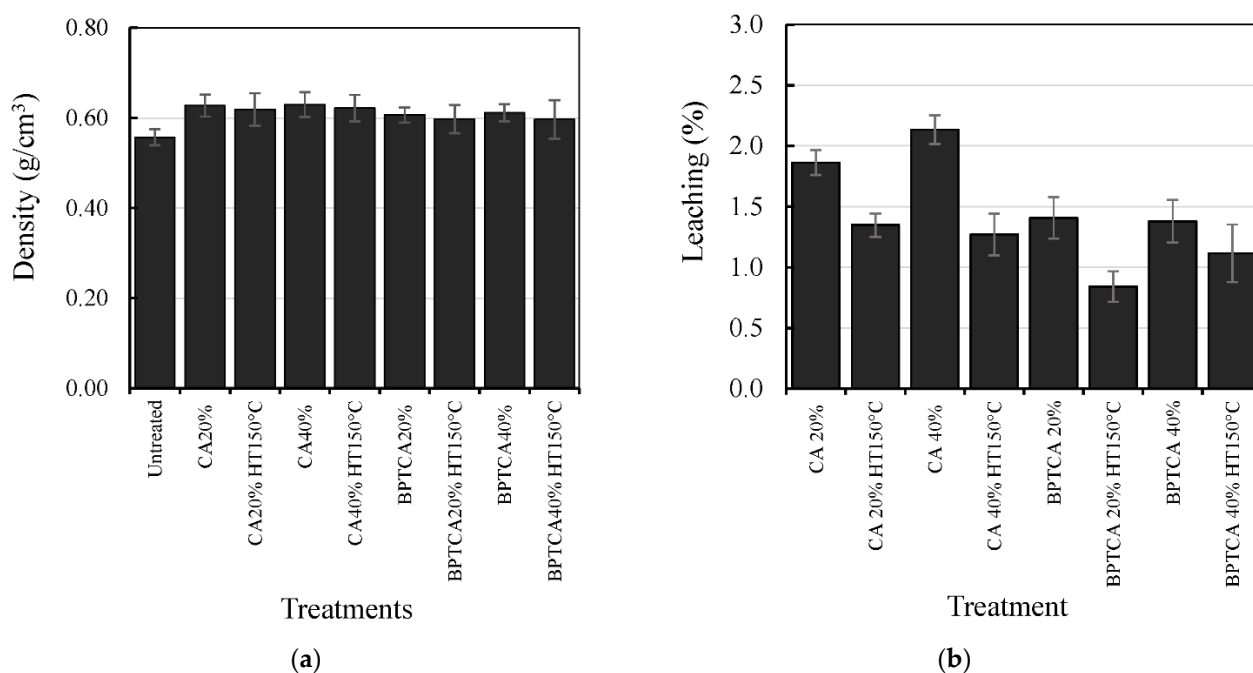
#### 3.1. Retention, WPG, Density, Leachability, and Dimensional Stability (ASE and WU)

The retentions of CA and BPTCA were an average of 24  $\text{kg}/\text{m}^3$  and 19  $\text{kg}/\text{m}^3$ , respectively. The results in Figure 1 indicate that the retention by 20% and 40% concentration was almost the same for both CA and BPTCA. The slightly lower retention of the BPTCA could be due to its higher molecular weight (226.23  $\text{g}/\text{mol}$ ) compared to that of CA (192.12  $\text{g}/\text{mol}$ ). However, these values were larger than the minimum retention of 8  $\text{kg}/\text{m}^3$  recommended for commercial preservatives used in conventional preservation treatment, such as copper chrome boron (CCB), for housing and building in Indonesia. The results in Figure 1 also show that the WPG of the teak wood treated by CA and BPTCA was on the average of 28% and 25%, respectively. The increases in the WPG were due to the CA and BPTCA filling cell cavities and the void spaces within the cell wall. Linearly correlated with the retention values, it seemed that the increase in concentration both for CA and BPTCA did not increase the WPG of the modified wood. The impregnability of the wood, the high concentration of the impregnating solution, and the impregnation system might explain the low retention and WPG values of the solution 40%. Nevertheless, a similar WPG value was also reported by Martha et al. [16]. It was noted in the study that treated teak wood presents a WPG value of around 20% after furfuryl alcohol treatment with 45% of solution concentration, the polymerization of furfuryl alcohol is identified, and chemical interaction occurs between furfuryl alcohol and lignin.



**Figure 1.** Retention and WPG of CA and BPTCA-treated teak wood without and with heat treatment at 150 °C.

Variations of density among the untreated and treated teak woods are presented in Figure 2a. The density of the untreated teak wood was  $0.56 \text{ g/cm}^3$  and of the treated wood was in a range between  $0.60$  and  $0.63 \text{ g/cm}^3$ . The increase in density of an average of  $5\%$  was caused by the increase in the WPG of  $26\%$ . The deposition of the CA and BPTCA in the cell cavities and in the void spaces within the cell wall increases the density and imparts new properties to the treated wood. It was reported in another study that furfuryl alcohol treatment improves the density of wood because its cell walls are filled with polymerized furfuryl alcohol [25]. In addition, the impregnation of wood with a polymer causes not only the penetration of the polymer but also strengthens the wood cell walls [6].

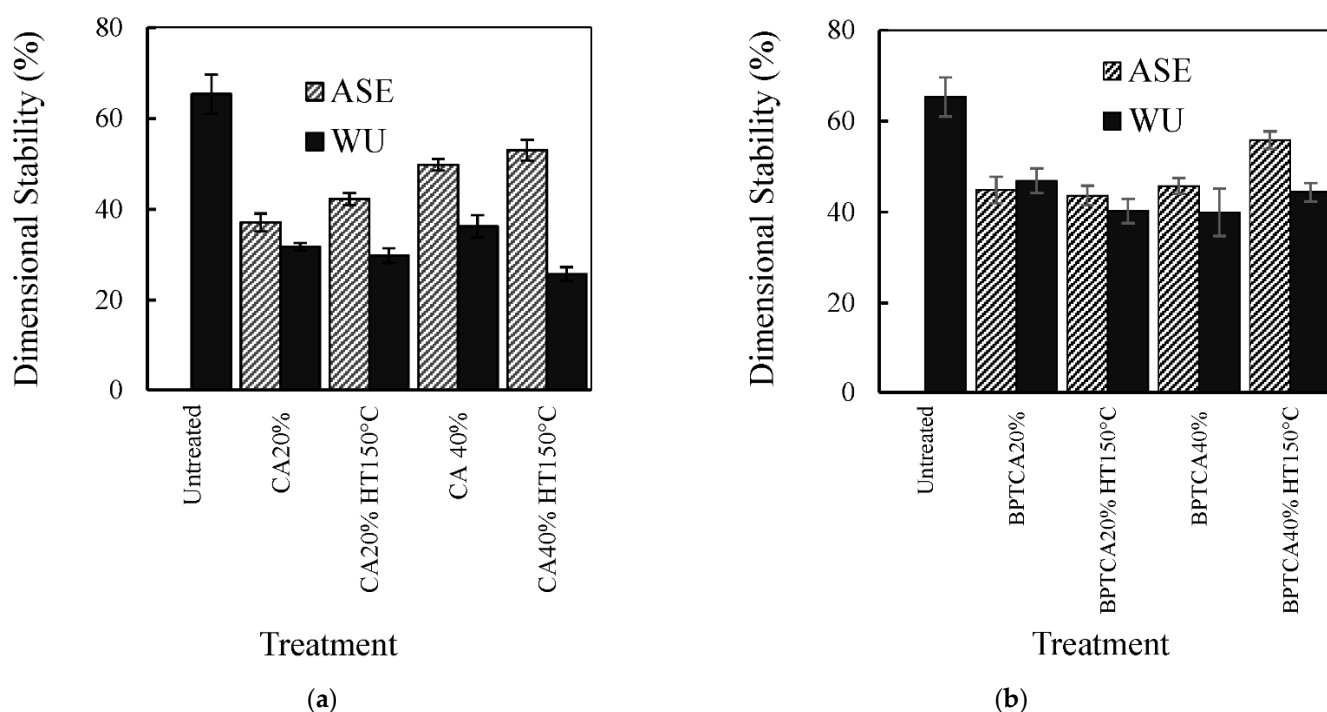


**Figure 2.** Variation of density (a) and leachability (b) among the untreated and treated SRT wood.

The results in Figure 2b show that the leachability of treated teak wood had an average of  $1.65\%$  for CA treatment and  $1.19\%$  for BPTCA treatment, which indicated a good fixation of CA and BPTCA in the teak woods. The results in Figure 2b also indicate that the values of leaching decreased slightly after the CA and BPTCA treatment followed by heating at  $150^\circ\text{C}$ . In another study, the leaching value was reported to be  $0.20\%$  for teak wood after glycerol-maleic anhydride treatment followed by heating at a higher temperature of  $220^\circ\text{C}$  [17]. This slightly lower leaching is caused by the occurrence of polymerization of the glycerol-maleic anhydride and lignin at  $220^\circ\text{C}$ .

The ASE and WU of the untreated and treated teak wood are shown in Figure 3. The increase in ASE value was followed by the decrease in WU value after CA and BPTCA treatments. ASE of CA and BPTCA-treated teak wood reached  $46\%$  and  $48\%$ , respectively. The results in Figure 3 indicate that remarkable differences in the value of ASE were not observed between unheated and heated CA and BPTCA. The increase in the ASE values suggested that the impregnation of the CA and BPTCA can improve the dimensional stability of the SRT wood. The interaction between CA or BPTCA and the wood components through esterification of the wood hydroxyl groups and a possible aromatic substitution of the wood lignin might explain the hydrophobic property of the modified teak, increasing the ASE values from the original wood. Further, the reduction in void volume in wood due to a possible lumen filling by CA and BPTCA polymer could also stabilize the teak wood dimension. Slightly higher ASE values were reported in other studies. ASE of furfurylated teak wood is an average of  $60\%$  [16], and ASE of glycerol-maleic anhydride unheated and heated at  $150^\circ\text{C}$  for teak wood is  $40\%$  and  $62\%$ , respectively [17]. ASE

of furfurylated beech wood containing 50% of furfuryl alcohol and 5% of tartaric acid is approximately 66% [11]. The WU value of the untreated teak wood was 65% and decreased to 31% after CA treatment and to 42% after BPTCA treatment. The WU decreases after CA and BPTCA treatment could be due to the less hydrophilic property of the modified wood as the results of the chemical reactions occurred between the wood components and the CA or BPTCA. The WU value for untreated teak is 94% and decreases to 37% after furfuryl alcohol treatment [16]. The WU of untreated teak wood is 92% and decreases to 52% after glycerol maleic anhydride and thermal treatment at 150 °C [17]. The decrease in the WU value indicates a reduction in wood hygroscopicity due to the decrease in free hydroxyl groups. The WU and ASE are also improved due to the decrease in crystallinity [26,27]. This may be attributed to the penetration of furfuryl alcohol into the amorphous region of the wood cell wall [26] and filling the empty cells as a bulking agent [28]. It could be considered that the CA and BPTCA came into the empty space in the cell wall as a bulking agents to replace water, then the treated teak wood dimension became more stable.

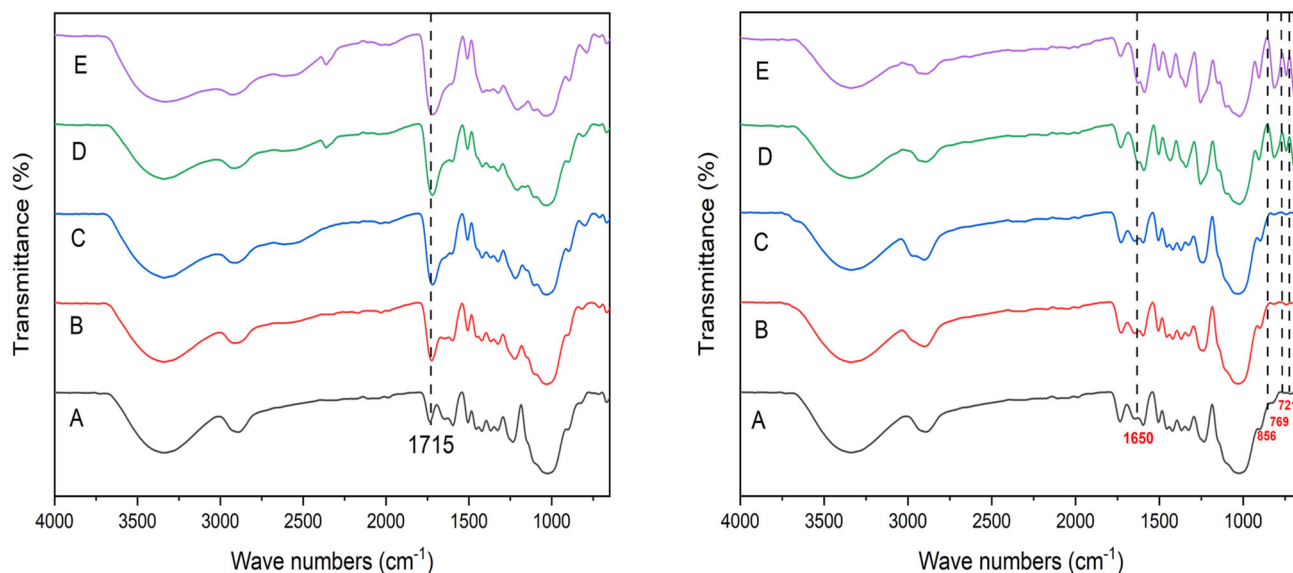


**Figure 3.** Dimensional stability (ASE and WU) of CA-treated (a) and BPTCA-treated (b) teak wood without and with heat treatment at 150 °C (HT150 °C).

### 3.2. Characterization of Impregnated SRT (FTIR and XRD Analysis)

Heating of the teak wood samples at 150 °C (HT150°C) after CA and BPTCA impregnation caused the occurrence of their chemical interaction with lignin, cellulose, or hemicellulose. Figure 4 (left) presents the FTIR spectra for the untreated, 20% CA, 20% CA followed by thermal heating at 150 °C, 40% CA, and 40% CA followed by thermal treatment at 150 °C over the 600 to 4000  $\text{cm}^{-1}$  wavenumber range. The absorption bands in the range of 3000–3600  $\text{cm}^{-1}$  are assigned as the stretching vibration of the hydroxyl group (-OH) in polysaccharides, which predominantly came from the celluloses and hemicelluloses in this case [29]. The FTIR spectra of all treatments in Figure 4 (left) indicated a relative decrease in intensity compared to untreated wood. The peak intensity decreased with increasing citric acid content and curing temperature. In the fingerprint area, the absorption bands in the spectra of untreated and treated woods were different. The spectra of treated teak presented the increase in the absorption band at 1715  $\text{cm}^{-1}$ , corresponding to the carbonyl stretching band, which presumably came from the ester group of CA-wood. The absorption differences in the lignin zone of around 1400–1600  $\text{cm}^{-1}$  were also identified, indicating

that there were also chemical interactions between CA and the lignin component of the teak, especially in the treatment using CA 40% at a temperature of 150 °C. Overall, the peak intensity increased along with increasing citric acid content from 20% to 40% and the increasing curing temperature. These observations confirmed that citric acid reacted with the hydroxyl groups of SRT wood to form ester linkages and contribute to reducing hygroscopicity.



**Figure 4.** FTIR spectra of untreated and CA treated (**left**), untreated and BPTCA-treated (**right**) of the short rotation teak wood. Note: A = untreated, B = 20% concentration, C = 20% concentration followed by thermal heating at 150 °C, D = 40% concentration, E = 40% concentration followed by thermal heating at 150 °C.

Figure 4 (right) shows the FTIR spectra of the untreated 20% BPTCA, 20% BPTCA followed by thermal heating at 150 °C, 40% BPTCA, and 40% BPTCA followed by thermal heating at 150 °C over the 600 to 4000  $\text{cm}^{-1}$  wavenumber range. The FTIR absorbance at around 3000–3600  $\text{cm}^{-1}$  for the treated wood, which was assigned as the stretching vibration of the hydroxyl group in polysaccharides, showed a relative decrease in intensity compared to untreated teak wood. The peak intensity of hydroxyl groups decreased with increasing BPTCA content and curing temperature. The decrease in the intensity of the hydroxyl groups caused the wood to be more hydrophobic, increasing the wood's dimensional stability (Figure 3). The absorption bands in the spectra between untreated and treated wood were different, especially in the fingerprint area. The absorption bands of the treated woods showed a difference after 40% BPTCA treatment and 40% BPTCA, followed by thermal heating at 150 °C. The increase in the absorption band at 1650  $\text{cm}^{-1}$  is associated with C=O stretching, which presumably occurred due to the interaction between BPTCA and the lignin component of the teak, leading to prominent differences in the lignin zone at around 1400–1600  $\text{cm}^{-1}$ . Three small bands were also observed at 856, 769 and 721  $\text{cm}^{-1}$  for 40% BPTCA treatment and 40% BPTCA followed by thermal heating at 150 °C. These small bands are assigned to aromatic C-H out-of-plane deformation [29]. After all, the different absorbance bands observed in the spectra of 40% BPTCA treatment and 40% BPTCA followed by thermal heating at 150 °C treatment indicates that the chemical interaction occurred between polycarboxylic acid and wood lignin.

Crystallinity analysis results by means of X-Ray Diffraction analysis of the modified woods are shown in Table 3. Crystallinity is defined as the degree of structural order of the molecules in a polymer [30]. In general, the crystallinity of the modified teak woods without heat treatment was higher than that after heat treatment and the teak woods

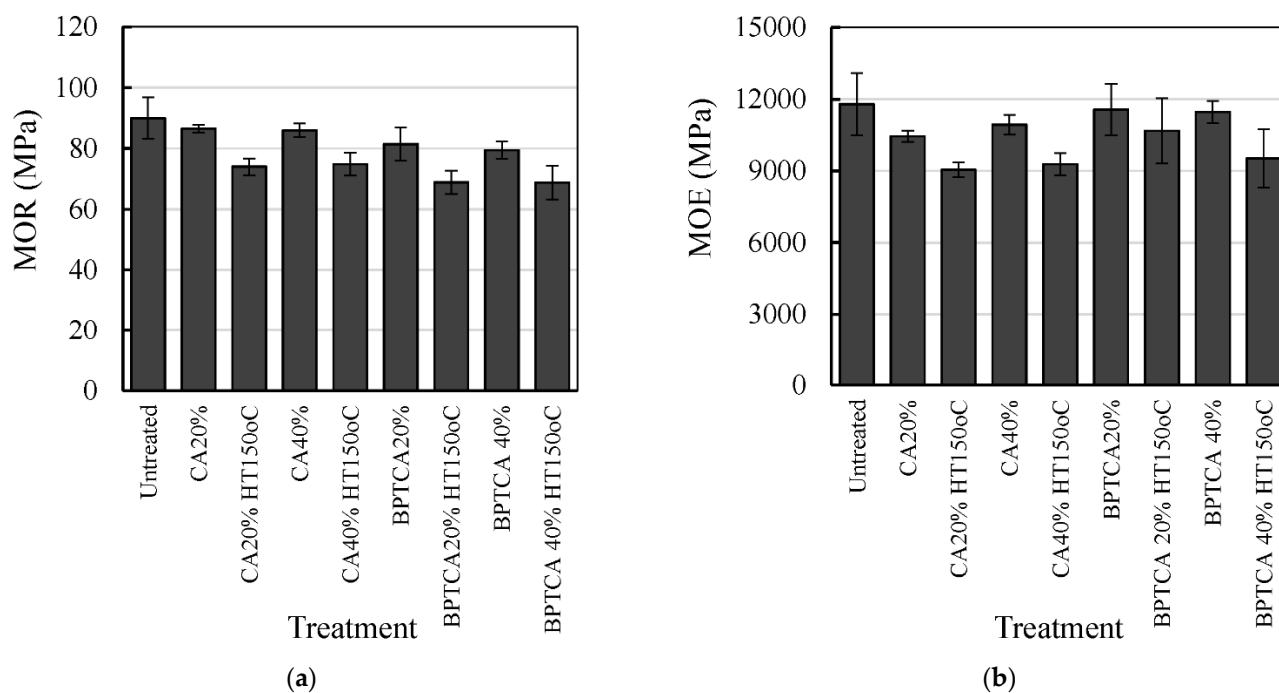
modified by BPTCA showed higher crystallinity than that of CA treatments. In a previous study, the decrease in crystallinity of jabon wood treated with a copolymer of melamine formaldehyde furfuryl alcohol (MFFA)-SiO<sub>2</sub> decreased compared to the untreated wood due to the grafting reaction of this additive with the wood components, especially in the amorphous region [31]. Similarly to this, since the impregnated chemicals in the current study were based on the weight percent, BPTCA with higher molecular weight presented a lower molar value than that of CA, hence the amount of possible grafting formation of BPTCA with the wood components was lower than that of CA treatment. Therefore, the degree of crystallinity of the BPTCA treatments was higher than CA treatments. On the other hand, although the untreated wood in this recent study was not tested, it is commonly known that the degree of the crystallinity of the thermally modified wood tends to increase with the increase in heat treatment temperature [32]. In harmony with the previous FTIR results above, heat treatment at 150 °C imparted the modified wood to have a lower degree of crystallinity due to the intensive interaction/reaction of BPTCA or CA with the wood components, generating new characteristics of the original teak wood.

**Table 3.** The crystallinity of 15-year-old short-rotation teak woods impregnated using CA (citric acid) and BPTCA (benzophenone tetracarboxylic acid) chemicals, without and 150 °C heat treatment.

| Treatment | Heat Treatment | Degree of Crystallinity (%) |
|-----------|----------------|-----------------------------|
| BPTCA 20% | -              | 53.18                       |
| BPTCA 40% | -              | 55.8                        |
| CA 20%    | -              | 45.93                       |
| CA 40%    | -              | 44.95                       |
| BPTCA 20% | 150 °C         | 38.95                       |
| BPTCA 40% | 150 °C         | 43.71                       |
| CA 20%    | 150 °C         | 38.94                       |
| CA 40%    | 150 °C         | 35.89                       |

### 3.3. MOE and MOR

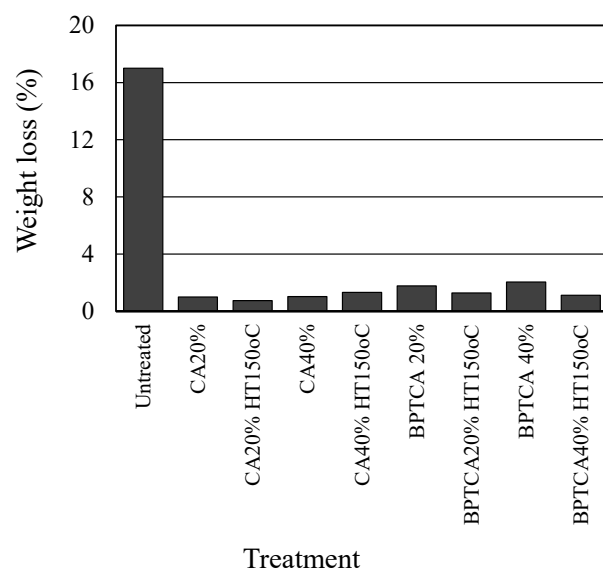
MOE and MOR of the untreated and CA and BPTCA-treated SRT are presented in Figure 5. The mean MOE values for untreated, CA, and BPTCA-treated samples were 11,769.13 MPa, 9947.04 MPa, and 10,807.50 MPa, respectively. This result indicates that the MOE decreased by 15% after CA was treated and 8% after BPTCA treatment. The values of MOE for the 20% and 40% of CA and BPTCA were almost the same. These similar MOE values could be due to the similar WPG values of the corresponding modified woods, which directly increase the wood density and predominantly effected the mechanical properties of the wood. However, the values of MOE of the CA and BPTCA-treated teak wood decreased slightly as the CA and BPTCA-treated teak wood were heated at 150 °C. It was noted that the MOE value of furfurylated teak wood decreases by 14% [16], and of glycerol maleic anhydride treated teak wood decreases by 3% [17]. The decrease in MOE value was also reported for furfurylated beech wood catalyzed by maleic anhydride [11]. The reason for the decrease in MOE has been clearly reported due to the depolymerization of wood polymers [11,16,17]. The MOR of untreated teak was 90 MPa, and the MOR values of CA and BPTCA-treated teak wood were 80.35 MPa and 74.64 MPa, respectively. This result indicates that the MOR decreased by 11% after CA was treated and 17% after BPTCA treatment. In the same phenomenon as in the MOE, the values of MOR for 20% and 40% of CA and BPTCA were almost the same. However, the values of MOR of the CA and BPTCA-treated teak wood decreased slightly as the CA and BPTCA-treated teak wood were heated at 150 °C. It was reported that the MOR of teak wood treated by glycerol maleic anhydride followed by heating at 150 °C decreased by 29% [17], and treatment using furfuryl alcohol followed by heating at 120 °C decreased 20% [16]. The decrease in MOR of the teak wood after CA and BPTCA can be explained due to the acidic property of the CA and BPTCA solution and of the heat imposed which caused the degradation of chemical compounds [16,17,33,34].



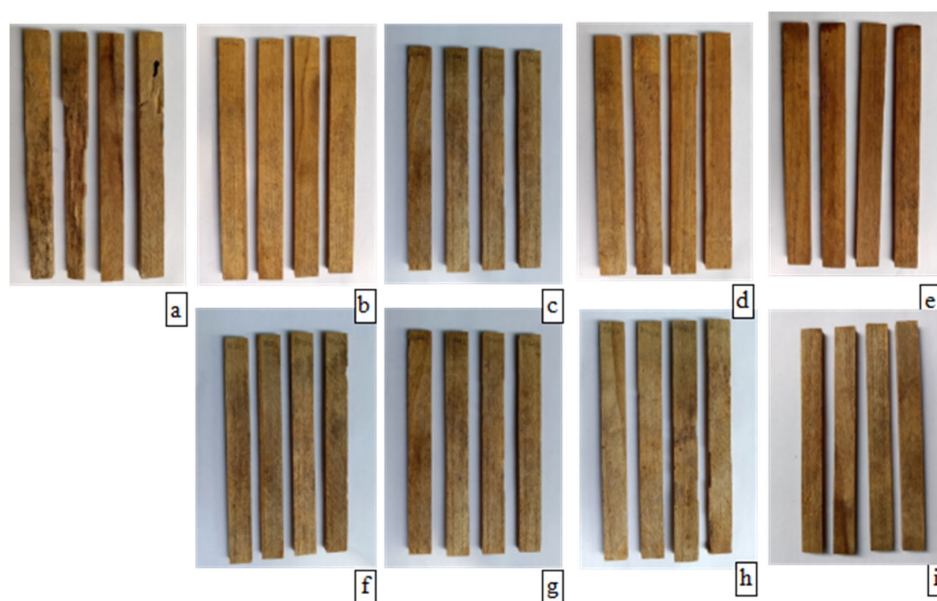
**Figure 5.** MOR (a) and MOE (b) values of CA and BPTCA-treated teak wood without and with heat treatment at 150 °C (HT150°C).

### 3.4. Durability against Subterranean Termites in the Field Test

Weight losses of untreated, CA and BPTCA-treated teak wood against subterranean termites were 17.00%, 1.03%, and 1.56%, respectively (Figure 6). The results in Figure 6 show that the concentration of 20% and 40% of both CA and BPTCA of unheated and heated samples generated almost the same weight loss against termite. This result indicates that the use of CA and BPTCA with or without thermal treatment could be valuable to protect the SRT wood against termites. The CA and BPTCA resulted in the fixation with wood components and/or wood degradation products formed, leading to a new material that was unable to be digested by termites. According to SNI 7207 [22], the termite resistance of untreated teak was classified as moderate (class III), and that of CA and BPTCA-treated teak wood was classified as very resistant (class I). The results in Figure 7 show the appearance of the untreated samples after three months tested in the field suffered severe damage along their cross-sections due to attack by termites. The damages on their sections reached 22% of their volume (Table 4). The damages were mostly on the buried part of the samples which indicated the attack of subterranean termites. The species of the subterranean termites were *Macrotermes sp.* and *Microtermes sp.* The damages of the CA and BPTCA-treated teak samples were due to slight attacks by the subterranean termites. The percentage of the damages on the CA and BPTCA-treated samples was less than 3%. According to the ASTM D 1758 [23], the termite resistance of untreated was classified as grade 7, while the of CA and BPTCA-treated teak wood was classified as 9–10 (Table 4). It was reported that the weight losses of untreated and glycerol maleic anhydride (GMA) treated teak followed by thermal treatment at 150 °C were 34.13% and 19.49%, respectively [17]. In addition, although the GMA treated teak at 150 °C produces better resistance against fungal decay, it suffers high mass loss against termites [17]. According to the SNI 7207 standard, the termite resistance of the GMA treated teak at 150 °C teak wood was classified as a very poor resistance (class V). This result indicates that the use of CA and BPTCA combined with thermal treatment could be valuable to protect the short rotation teak wood against termites.



**Figure 6.** Weight loss of untreated, CA and BPTCA-treated teak wood without and with heat treatment at 150 °C (HT150 °C).



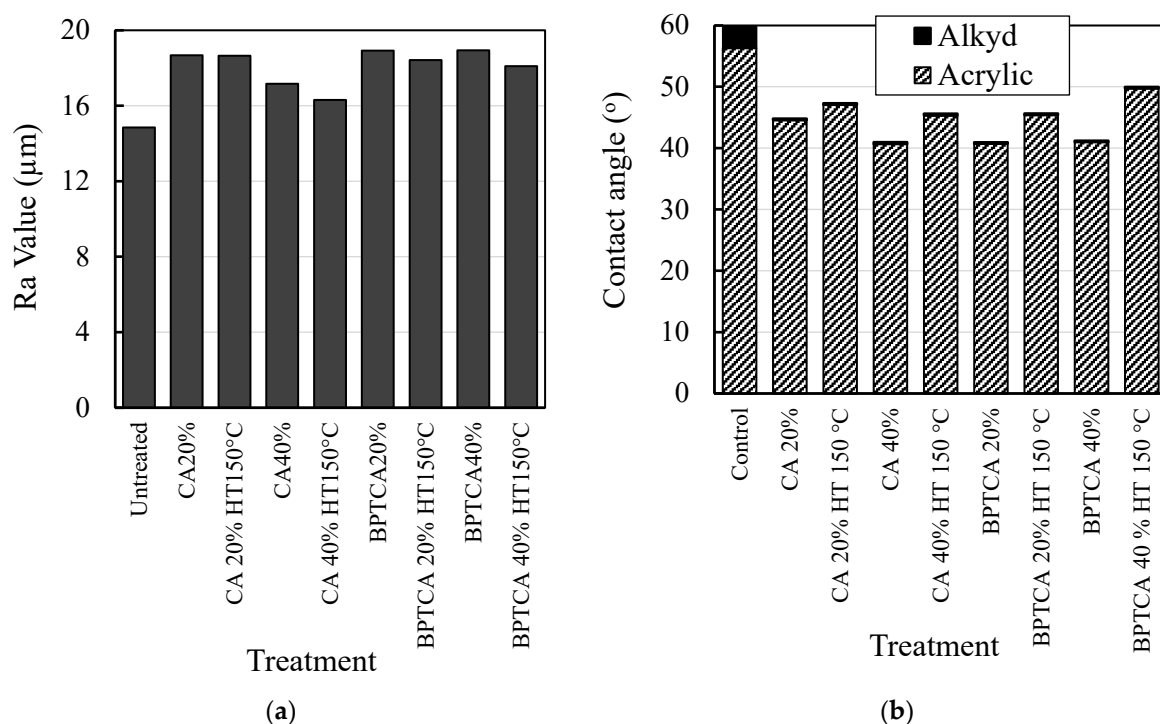
**Figure 7.** The appearance of untreated and treated teak wood samples after 3 months of graveyard test. Note: (a) = untreated, (b) = CA20%, (c) = CA20% HT150°C, (d) = 40%CA, (e) = 40%CA HT150°C, (f) = BPTCA20%, (g) = BPTCA20% HT150°C, (h) = 40%BPTCA, (i) = 40%BPTCA HT150°C.

**Table 4.** Durability rating of the untreated and treated woods based on a percentage of damage of stakes attacked by subterranean termites.

| Treatments       | Percentage of Damages (%) | Rating |
|------------------|---------------------------|--------|
| Untreated        | 22.7                      | 7      |
| CA20%            | 0                         | 10     |
| CA20% HT150°C    | 0                         | 10     |
| CA40%            | 0.44                      | 9      |
| CA40% HT150°C    | 0.66                      | 9      |
| BPTCA20%         | 1.49                      | 9      |
| BPTCA20% HT150°C | 1.49                      | 9      |
| BPTCA40%         | 1.18                      | 9      |
| BPTCA40% HT150°C | 0.54                      | 9      |

### 3.5. Surface Roughness and Wettability

Figure 8 shows the variation of surface roughness as presented by the arithmetical mean roughness ( $R_a$ ). The  $R_a$  values of untreated, CA, and BPTCA-treated teak wood were an average of  $14.85\ \mu\text{m}$ ,  $17.69\ \mu\text{m}$ , and  $18.59\ \mu\text{m}$ , respectively. The slightly higher roughness of the CA and BPTCA-treated teak wood could be important in determining their wettability for water-based acrylic and oil-based varnishes wettability. The values of the contact angle of the water-based acrylic and oil-based alkyd are shown in Figure 8. The untreated teak wood with a slightly lower  $R_a$  value generated a higher contact angle of  $56.29^\circ$  for acrylic varnish and  $3.46^\circ$  for alkyd varnish. The contact angle of the CA and BPTCA-treated teak woods were almost the same with an average of  $44.31^\circ$  for acrylic varnish, and  $0.24^\circ$  for alkyd varnish. These results indicate that the CA and BPTCA-treated teak wood tended to provide a lower contact angle. The treated teak woods with a lower contact angle caused the water-based acrylic and oil-based alkyd varnish liquid to spread and penetrate easier in their surfaces. Fortunately, the CA and BPTCA-treated teak woods in this work presented water-based acrylic and oil-based alkyd contact angle values lower than  $90^\circ$ , which indicate their high wetting ability against the varnish liquids.



**Figure 8.**  $R_a$  (a) and Contact angle (b) of untreated, CA and BPTCA-treated teak wood without and with heat treatment at  $150\ ^\circ\text{C}$  (HT150  $^\circ\text{C}$ ).

Considering the results presented above, the treated SRT wood should be expected to exhibit remarkable dimensional stability, wettability, and durability compared to other wood species. Its higher stability and termite resistance would guarantee its utilization to some extent. The treated SRT should be superior to many other timbers of fast-growing plantations such as Indonesian Sengon (*Paraserianthes falcataria*) and Jabon (*Anthocephalus cadamba*) [35], and could be homogenized with long rotation teak wood, which is widely used. The future for SRT wood modification by thermal and chemical methods looks very promising. There are already many new wood substitutes for solid-treated wood, including engineered wood composites such as wood-plastic composites and products such as preserved oriented strandboard (OSB), laminated veneer lumber (LVL), and parallel strand lumber (PSL). All of these products will require both new and existing preserving technologies to prevent colonization by decaying organisms and infestation by wood-

destroying insects. The evaluation of engineered wood composites already in service has demonstrated that those without preservation have exhibited both decaying and fruiting bodies after exterior exposure.

#### 4. Conclusions

The non-biocide of citric acid (CA) and benzophenone tetracyclic acid (BPTCA) allows for the improvement of the properties of short rotation teak (SRT) wood. The dimensional stability of the CA and BPTCA-treated teak wood is increased, though the MOE and MOR are slightly decreased. Durability against subterranean termites of the CA and BPTCA-treated SRT wood is classified as very durable (class I and grade 9 to 10). FTIR and XRD analysis indicate that the chemical modification occurred between CA and BPTCA with the teak wood cell wall polymers. The treatments of CA and BPTCA produce good wettability of both acrylic and alkyd varnishes on the teak wood surface, promoting a good bonding quality of varnish and paint. The CA and BPTCA treatments are expected to be eco-friendly and valuable methods for improving the quality of SRT for high-quality wood product utilization for both interior and exterior purposes. Accordingly, for a complete durability study against biological degrading agents, a decay durability test against fungi (white rot, brown rot, and soft rot) would be performed as part of our future work.

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