Book Chapter

Performance in Accelerated Laboratory Tests of Oil Heat Treated 16-Year-Old *Acacia mangium*

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Abstract

Performance of the durability of oil heat-treated 16-year-old *Acacia mangium* through accelerated laboratory tests were studied. *A. mangium* logs of known age harvested and segregated into the bottom, middle, and top portions. These were oil-heat treated in a tank with oil palm oil as a heating medium at temperatures 180, 200 and 220°C for the duration of 30, 60 and 90 minutes. The wood samples dried and grounded into sawdust were air-dried again before undergoing tests. An accelerated 12 weeks of laboratory durability studies conducted on the treated *A. mangium.* Fungi of *Pycnoporus sanguineus*, *Gloeophyllum trabeum* and *Coriolus versicolors* inoculated on the woods. Untreated samples used as controls. The results showed that the durability of the wood increases with an increase in temperature and duration of the treatment. The hot oil-treated samples could reduce the attack of *G. trabeum* from 5.02%, 4.41% and 4.38% in the control samples to 0.54-4.55%, 0.91-4.41% and 1.08-4.38% at the bottom, middle and top portions, respectively. The attack of *C. versicolors* reduced from 11.48%, 14.27% and 15.68% in the control samples to 1.87-10.19%, 3.10-12.69 and 4.78-15.10% at the bottom, middle and top portions, respectively. However, the attacked of *P. sanguineus* were less effective with 31.42%, 18.24% and 10.53% in control samples to 3.71-10.18%, 5.74-14.59% and 4.37-17.08% at the bottom, middle and top portions, respectively. Massive colonization of mycelia occurs in vessels of the untreated *A. mangium* wood in comparison to the oil heat-treated wood observed through the scanning electron microscope.

Keywords

Acacia mangium; Durability; Fungi Inoculation; Oil Heat Treatment; Scanning Electron Microscope

Introduction

Ease of growth and adaptability has made *Acacia mangium* one of the most popular plantation species in Malaysia. This fast wood growing species, however, has some disadvantage in

which the wood contains a high proportion of juvenile wood and poorly developed heartwood particularly at the ages between 14 to 18-year-old [1]. It has wide growth rings that produce low-density wood which exhibited inferior mechanical properties. The durability of the wood becomes weak due to these problems resulting in the wood being attacked by bio-deteriorating agents [2]. The wood of *A. mangium* needs treatment to enhance its properties and durability. The oil heat treatment process can be applied to improve *A. mangium* durability [3,4]. The heat treatment is one of the wood modification processes used in the wood industry compared to other processes [3]. This treatment is considered an environmental-friendly process it does not uses chemicals [5,6].

Materials and Methodology

Tree Harvesting

Three (3) defects free 16-year-old *Acacia mangium* trees harvested from Batu Melintang, in Kelantan. The tree selected based on its excellent form, long straight trunk, decay-free and with minimum branches. The age of the trees were determined measuring their diameter at breast height (DBH) and the tree height. The DBH of the harvested *A. mangium* were 30 cm with tree height of 12 m. The tree felled using a chainsaw and segregated into the bottom, middle and top portions. The logs then transported to Universiti Malaysia Kelantan (UMK) for the subsequent process.

Sample Preparation

The woods containing both heartwood and softwood cut into sized 30 cm (L) x 10 cm (W) x 2.5 cm (T) using a chainsaw and a table saw.

Oil Heat Treatment Process

The wood was oil heat-treated in a stainless steel tank using palm oil as the heating medium (Figure 1). The treatment uses three (3) different temperatures 180^{o}C, 200^{o}C and 220^{o}C and

three (3) treatment durations 30, 60 and 90 minutes. Three replicates of woods used for each treatment. Altogether 81 wood samples of sizes 30 cm x 10 cm x 2.5 cm were treated. Method outlines by Izran *et al*., [7]; Izyan *et al.*,[5]*;* Rafidah, [8]; Razak *et al.*, [9] used in the studies. The wood then removed from the tank after each treatment period ended. Excessive oil on the surface of the sample wiped with a clean cloth as steps to prevent excessive oil from entering the wood tissue. All the samples were then cooled down and stored in the conditioning chamber at 25±2^{o}C, and relative humidity (RH) of 65±5% before undergoing the testing procedure. Once the moisture contents of the wood reach 12%, they were taken out for resizing and tested for basic density and 12 weeks accelerated laboratory durability tests.

Figure 1: Wood blocks of *Acacia* hybrid (*ca*. 55 x 10 x 3 cm) soaked into palm oil in a heatable tank to test treatment effects on wood durability.

Density

Determination of the density of each sample tests conducted according to ISO 3131-1975 standard (Wood: Determination of

Moisture Content for Physical and Mechanical Tests). The samples of treated and untreated prepared into a size 2 cm x 2 cm x 2 cm. Then, samples weighed by using the analytical balance, and initial volumes (length x width x thickness) of wood measured by using a digital vernier calliper. The samples were then dried in the oven at 103±2ºC at least for 24 hours or until the moisture reached 12%. Before weighing the samples to get their final weight, all the samples kept in the desiccators for 15 minutes. After that, the final weight and volumes of samples were measured and recorded.

Durability Test for Treated and Untreated *Acacia mangium*

Durability testing against three (3) wood-decaying fungi (*Pycnoporus sanguineus*, *Gloeophyllum trabeum*, and *Coriolus versicolors*) has been conducted to compare the strength of wood before and after the heat-treatment process. The duration of this durability test was 12 weeks, and the standard used in this testing based on the American Wood Preservatives Associations Standard [10] – M10-77 (Standard Method of Testing Wood Preservatives from Soil-Block Culture).

Preparation of Culture Media

The cultured media that used in this study was Potato Dextrose Agar (PDA) that supplies the nutrient for fungi to growth as represented in Figure 2. 15 g of PDA was weighed and put into 250 ml Duran bottle. The agar dissolved in distilled water. Then, PDA agar had been sterile by using an autoclaved machine at 15 psi for 20 minutes at 120ºC. The agar was then poured into a petri dish, sterilized Duran bottle and left to be cool. The culture media that prepared in a petri dish used for inoculation of pure fungal culture whiles the media in Duran bottle were for durability test. Culture media prepared in Duran bottle should be left about 45º to increase the surface of media.

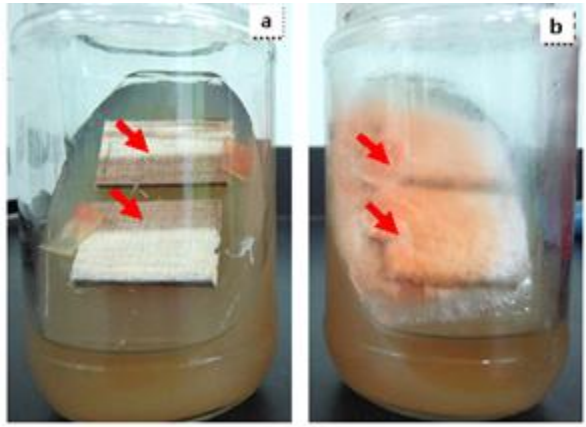

Figure 2: Culture media (MEA) with feeder strips of *Acacia* hybrid woods and mycelial plugs of the test fungi, *P. sanguineus* (arrows) in 250 ml Duran bottle (a) was fully covered by mycelia after 3 weeks incubation (b) at 26.7°C and 70% RH in darkness.

Preparation of Test Blocks

Sterilized samples sized 2.5 cm x 2.5 cm x 0.9 cm were used for durability test. Each wood samples were wrapped with aluminium foils and autoclaved at 15 psi for 20 minutes. After cooling and dried in laminar flow, the samples aseptically placed onto the culture of decay fungi by using the scalpel.

Inoculation of Fungi

The fungi *P. sanguineus, G. trabeum*, and *C. versicolors* used in this study obtained from the Forest Research Institute of Malaysia (FRIM). The inoculation process conducted in a laminar flow and cleaned by using the 75% ethanol. The apparatus used were all sterile. Fungi were placed upside down on the media in the Duran bottle. These done properly as not to damage the fungi. The bottles stored in an incubator set at 37ºC and left for 12 weeks for the fungi to grow. *P. sanguineus* and *C. versicolor* were the white-rot fungi. The illustrated of the process were represent in Figure 3 (a, b, c).

(A)

(B)

(C)

Figure 3: An inoculation process represented on a, b, and c, respectively; (a) fungi were taken using a borer (b) upside down fungi onto new media (c) fungi on the new media.

Measurement of Weight Loss

After 12 weeks, the *A. mangium* wood blocks removed from the bottled. The samples cleaned by using the scalpel and brush to remove mycelium. Then, all the samples were put into the oven at 60±5ºC until the blocks reached its constant weight.

Micromorphology of Colonized *Acacia mangium*

After 12 weeks of exposure, the samples of *A. mangium* cleaned from mycelia by using a brush. Then, the samples were cut into sized 0.3 cm x 0.3 cm x 0.3 cm. The structure of these samples analyzed by scanning electron microscopy (SEM) for hyphal colonization of the woods.

Results and Discussion

Density

Table 1 shows the results for density for each sample under different temperature and duration of treatment at 12% MC. The untreated wood used as a control. The density at the bottom and middle portions were 0.53 g/cm³ respectively compared to the top portion 0.52 g/cm³.

Table 1 showed the density of treated samples increased when treated at 180 and 200°C for 30 to 90 minutes. The densities decrease in values after treated at 220°C for 60 and 90 minutes. All samples taken at the bottom, middle and top portions exhibited the same trends. The increased in the wood density might be due to the oil that able to penetrate into the cell wall and influence the overall total weight of the sample [11] and resulted in the thickening of the cell wall [12]. The density of wood then started to decrease when treated at high temperature for a long duration of time. These might be due to changes in the wood structure after the treatment as the results of the removal of wax, resin, and others that occur during the treatment [13]. The removal of those substances from the wood structure decreases the density of wood that can slightly reduce the strength of wood.

Table 1: Mean values of *Acacia mangium's* density at 12% moisture content.

Temperature (ºC)	Treatment duration (min)	Density (g/cm^{-3})		
		Bottom	Middle	Top
0 (Control)	0 (Control)	0.53	0.53	0.52
	30	0.55 (+3.78)	0.54 (+1.89)	0.53 (+1.92)
180	60	0.54 (+1.89)	0.54 (+1.89)	0.53 (+1.92)
	90	0.54 (+1.89)	0.54 (+1.89)	0.53 (+1.92)
	30	0.58 (+9.43)	0.55 (+3.77)	0.53 (+1.92)
200	60	0.57 (+7.55)	0.54 (+1.89)	0.54 (+3.85)
	90	0.54 (+1.89)	0.52 (+1.89)	0.53 (+1.92)
	30	0.54 (+1.89)	0.54 (+1.89)	0.53 (+1.92)
220	60	0.53 (0.00)	0.53 (0.00)	0.52 (0.00)
	90	0.53 (0.00)	0.53 (0.00)	0.52 (0.00)

Note: ( ) = % change from control

Durability of *Acacia mangium* Wood

The results in Table 2, showed the mean weight loss of *A. mangium* after exposed to the tests fungi. The fungi attack dropped when the temperatures and treatment duration increases as highlighted in Figure 4. This results proved that the oil-heat treatment process could apply to reduce the fungal attack of *A. mangium* woods. The oil-heat treatment increased the durability of *A. mangium* wood in all the portions. The top portions of *A. mangium* experienced the highest weight loss when exposed to all the fungi. The weight loss was 17.08% when exposed to *P. sanguineus*, 15.20% to *C. versicolor* and 5.72% to *G. trabeum* at temperature 180°C for 30 minutes. The lowest was 3.71% when exposed to *P. sanguineus*, 1.87% to *C. versicolor* and 0.54% to *G. trabeum* at temperature 220°C for 90 minutes for all bottom samples. The weight loss of samples taken from the middle portions showed values in between the bottom and top portions.

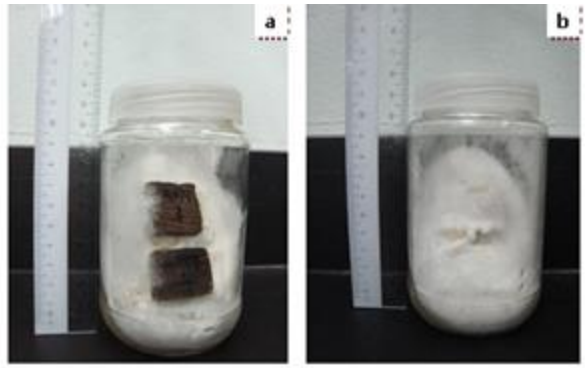

Figure 4: Oil-heat treated and untreated of *Acacia* hybrid wood blocks (*ca*. 2.5 x 2.5 x 0.9 cm) were exposed to the decay fungi (*C. versicolor*; a), and the mycelium (arrow) was covered the woodblocks after 12 weeks testing period (b).

The same results also obtained in the middle and bottom portions for the wood exposed to these three fungi. In the middle portions, the highest weight loss obtained from the wood that undergoes treatment one which is 14.59% for *P. sanguineus*, 12.69% for *C. versicolor* and 4.41% for *G. trabeum*. All the samples in treatment one had been exposed to the fungi after being treated at 180°C for 30 minutes. The lowest weight loss in the middle portion resulted from the wood that is treated in treatment 9 (T9) which are 5.74% (*P. sanguineus*), 3.10% (*C. versicolor*) and 0.91% (*G. trabeum*). While the lowest weight loss in the bottom portion is 3.71% (*P. sanguineus*), 1.87% (*C. versicolor*) and 0.54% (*G. trabeum*) that found in wood samples that treat at 220°C for 90 minutes.

The fungal attack, according to the fungi type and portions illustrated in Figures 5, 6, and 3, respectively presents wood durability against *P. sanguineus*, *C. versicolor*, and *G. trabeum*. Oil heat-treated *A. mangium* shows decreasing in overall of fungal attack. The oil-heat treatment had changed the components of wood. At a certain temperature and duration, the wood components (cellulose, hemicelluloses, and lignin) degraded at a different level. The degradation of those components depends on the temperature and duration of the heating process and wood species (Callum, 2006). This

modification in the wood makes them more resistance to the fungi attack.

The untreated *A. mangium* experiences the highest weight loss by the *P. sanguineus,* which is 10.53%-18.73% followed by *C. versicolor* (11.48%-15.69%) and *G. trabeum* (5.02%-5.72). *A. mangium* wood at bottom portions possesses the highest resistance against fungi attack compared to the top and middle portions. These is due to the high density of the wood at the bottom compared to the other portions [14]. The treatment is effective in controlling *G. trabeum* attacking the *A. mangium* woods. The attack of *G. trabeum* reduced from 5.72% (top), 5.22% (middle) and 5.02% (bottom) in control samples to 1.08%-4.38%, 0.91%-4.41%, and 0.54%-4.55%at top, middle and bottom respectively. The attacks by *C. versicolor* reduced from 15.69%, 14.27%, and 11.48% in the controlled samples to 4.78%-15.20%, 3.10%-12.69%, and 1.87%-10.19% at the top, middle and bottom portions. The treatment, however, was least effective to control the *P. sanguineus* attacks as the weight loss were 10.53%, 18.24%, and 18.73% in control samples to 3.71%-10.18%, 5.74%-14.59%, and 4.37%-17.08% at the bottom, middle, and top portions.

The oil-heat treatment proved effective in enhancing the durability of *A. mangium* wood [9,11]. The decreasing weight loss of *A. mangium's* wood by the fungi, however, are dependent on the temperature and duration of treatment. The decay fungi need suitable moisture and temperature to growth [15]. The white-rot fungi decomposed all the structural component of wood, especially lignin. It also has a complete cellulose complex that can degrade the lignin content in wood [15]. When the lignin had degraded after treatment given, the wood durability could increase because it would decrease the fungus attack since the lack of wood component [16]. The brown rot fungi used in this study is *G. trabeum* which attack the wood and decrease the weight loss of wood. These is because it decomposes the carbohydrates which are hemicelluloses and cellulose that obtained in the wood [15]. The decreasing of weight loss is between 40% and 50% in untreated samples to 36% and 15% after 12 weeks exposure to the *G. trabeum* and *C. versicolor*.

Table 2: Mean weight loss of *Acacia mangium.*

Treatment	*P. sanguineus*	*C. versicolor*	*G. trabeum*
Top Portion			
Control samples	18.73	15.69	5.72
T1 (180°C/30 min)	17.08 (8.81)	15.20 (3.12)	4.38 (23.43)
T2 (180°C/60 min)	15.42 (17.67)	15.08 (3.89)	3.36 (41.26)
T3 (180°C/90 min)	13.67 (27.02)	12.86 (8.04)	3.36 (41.26)
T4 (200°C/30 min)	14.87 (20.61)	11.18 (28.74)	3.65 (36.19)
T5 (200°C/60 min)	13.66 (27.07)	9.48 (39.58)	3.53 (38.29)
T6 (200°C/90 min)	13.31 (28.94)	8.77 (44.10)	2.54 (55.60)
T7 (220°C/30 min)	6.74 (60.01)	10.09 (35.69)	2.21 (61.36)
T8 (220°C/60 min)	5.71 (69.51)	5.96 (62.01)	1.66 (70.98)
T9 (220°C/90 min)	4.37 (76.67)	4.78 (69.53)	1.08 (81.12)
Middle Portion			
Control samples	18.24	14.27	5.22
T1 (180°C/30 min)	14.59 (20.01)	12.69 (11.07)	4.41 (15.52)
T2 (180°C/60 min)	11.67(36.02)	8.86 (37.91)	3.79 (27.40)
T3 (180°C/90 min)	8.98 (50.77)	4.48 (68.61)	3.23 (38.12)
T4 (200°C/30 min)	9.09 (50.16)	8.79 (38.40)	3.08 (41.00)
T5 (200°C/60 min)	8.49 (53.45)	5.68 (60.20)	2.56 (50.96)
T6 (200°C/90 min)	6.80 (62.72)	5.52 (61.32)	2.51 (51.92)
T7 (220°C/30 min)	7.08 (61.18)	7.94 (44.36)	2.83 (45.79)
T8 (220°C/60 min)	7.41 (59.30)	6.75 (52.70)	1.81 (65.33)
T9 (220°C/90 min)	5.74 (68.53)	3.10 (78.28)	0.91 (82.57)
Bottom Portion			
Control samples	10.53	11.48	5.02
T1 (180°C/30 min)	10.18 (3.32)	10.19 (11.24)	4.55 (9.36)
T2 (180°C/60 min)	9.30 (11.68)	6.31 (45.03)	3.91 (22.11)
T3 (180°C/90 min)	8.28 (21.37)	4.21 (63.33)	2.73 (45.62)
T4 (200°C/30 min)	9.18 (12.82)	6.83 (40.51)	2.76 (45.02)
T5 (200°C/60 min)	7.35 (30.20)	5.81 (49.39)	1.71 (65.94)
T6 (200°C/90 min)	4.24 (59.73)	4.87 (57.58)	1.11 (77.89)
T7 (220°C/30 min)	7.67 (27.16)	7.87 (31.45)	1.36 (72.91)
T8 (220°C/60 min)	4.65 (55.84)	7.00 (39.02)	0.66 (86.85)
T9 (220°C/90 min)	3.71 (64.77)	1.87 (83.71)	0.54 989.24)

Note: ( ) = % change from control

As the woods undergo the oil-heat treatment, the moisture content of the wood is decreasing. Moisture is the key ingredient for all types of biological damage of wood [17]. So, due to this condition, the fungi attack also fall because fungi need the moisture to attack the wood [15]. Active decay fungi attack could be stopped by removing the moisture since fungi become dormant when moisture content decrease below than 20% and the sorption of water into material limited because of the increased of hydrophobic character of wood which is not favorable to the growth of fungi [18,19].

Analysis of Variance and Correlation Analysis on Durability of treated *A. mangium*

Table 3 shows the analysis of variance (ANOVA) for physical of *A. mangium*. The analysis was conducted to determine whether there was a significance difference between the durability of wood with treatment temperatures, duration, and portion. Based on the table, there were significant differences between weight loss with the treatment temperatures (180°C, 200°C, 220°C) as a factor. These means that temperature of the treatment is affected the durability of *A. mangium* wood. As the duration of the treatment also affecting the strength of wood that studied in this research. While Table 4 highlighted that the correlation analysis of weight loss in oil heat treatment of *Acacia mangium*.

The wood height consisting of three portions shows a significant difference between weight loss and the portions. There is also a significant difference for the fungi variables which include three types of fungi. These outcomes also supported by research carried out by Razak *et al*. [1].

Table 3: ANOVA of durability of *A. mangium.*

S.V.	Sum of Squares	Df	Mean Square	F-Ratio
Temperature	678.676	2	333.338	20.534*
Duration	352.425	2	176.212	9.852*
Fungi	1994.747	2	997.374	90.325*
Portion	413.478	2	206.726	11.726*

** = significant at p≤0.01, * = significant at p≤0.05, ns = not significant.

Table 4: Correlation analysis of weight loss in the oil heat treatment of *A. mangium.*

	Portion	Density	Temp.	Duration	Fungi	Weight Loss
Portion	1.0000	5.0038e-001	0.0000	0.0000	0.0000e+00	0.2945
Density		1.0000	-0.2723	-0.4117	-9.3762e-018	0.0931
Temperature			1.0000	0.0000	0.0000e+00	0.3822
Duration				1.0000	0.0000e+00	0.2754
Fungi					1.0000	0.6215
Weight Loss						1.0000

Micromorphology of Colonized *A. mangium*

The micromorphology of the oil heat treatment of 18-year-old *A. mangium* wood observed to detect changes occurred before and after being exposed to the fungi. Observations made by using the Scanning Electron Microscope. The vessels structure were focussed into since they were the primary structure found in the wood for observation between untreated and treated wood. Figure 5 showed the images of untreated and treated *A. mangium* that being exposed to the *P. sanguineus*.

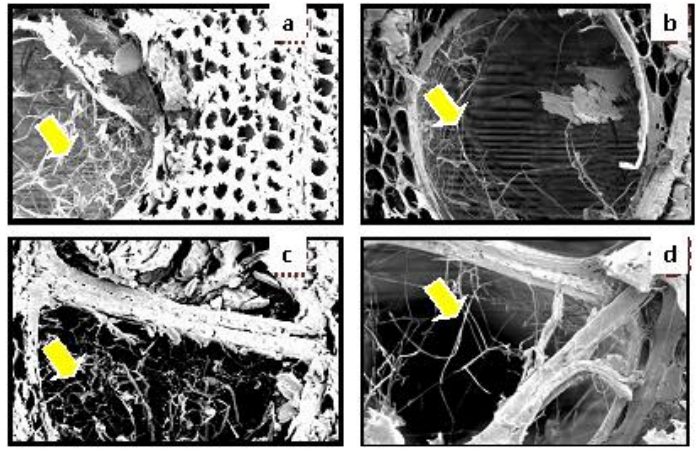

Figure 5: SEM images (transverse sections) of *Acacia mangium* woods sampled colonized by *P. sanguineus* after 12 weeks incubation period. A more prolific mycelia (arrows) permeating and filling the lumen of vessels of untreated wood (a, c) was observed as compared to wood experienced Treatment 9 (b, d).

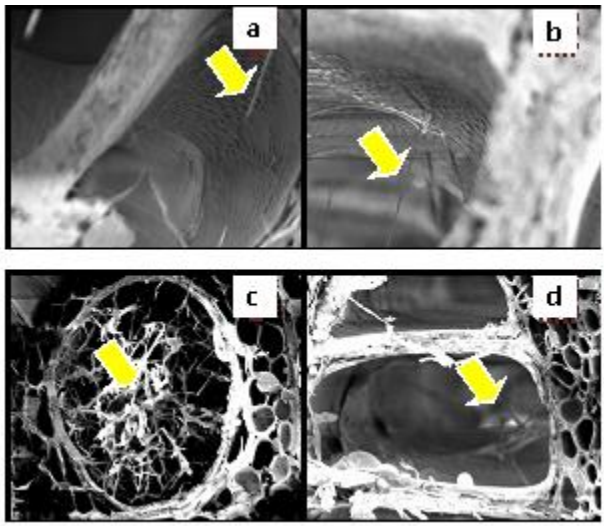

Figure 6: SEM images (transverse sections) of *Acacia mangium* woods sampled colonized by *C. versicolor* after 12 weeks incubation period. More prolific mycelia (arrows) permeating and filling the lumen of vessels of untreated wood (a, c) was observed as compared to wood experienced Treatment 9 (b, d).

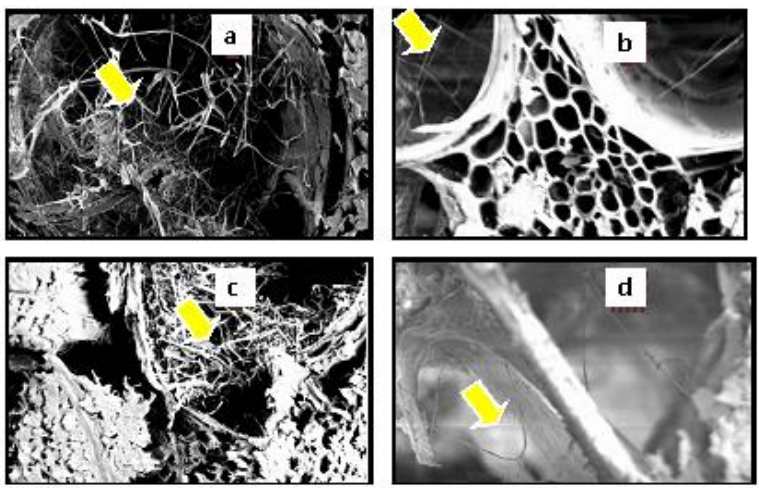

Figure 7: SEM images (transverse sections) of *Acacia mangium* woods sampled colonized by *G. trabeum* after 12 weeks incubation period. More prolific mycelia (arrows) permeating and filling the lumen of vessels of untreated wood (a, c) was observed as compared to wood experienced Treatment 9 (b, d).

The wood treated at 220°C for 90 minutes shows the less colonization of mycelia compared with the untreated wood. The samples exposed to the fungi for 12 weeks. The same pattern of results also obtained for the wood that exposed to the *C. versicolor* (Figure 6) and *G. trabeum* (Figure 7). From the images, it can seem that there were changes that occur after the wood samples treated by using the oil-heat treatment. The untreated *A. mangium* wood shows the massive colonization of mycelia within the wood vessel compared with the treated wood. These showed that the oil-heat treatment had affected the structure of wood.

The oil heat-treated wood experience changes in structure. Modification of the cell wall structure, causing the deformation of the wood cells, especially the vessels. Razak *et al*., [14] also came out with the same pattern of results where hyphae and mycelia could observe within all cell types such as vessels, fibres and parenchyma. Vessels were the most resistant elements apparently to degradation by fungi. The factor of the massive colonization of mycelia in untreated samples compared with oil-heat treated was because during oil-heat treatment process the hydrophobic character of wood has increased which caused limited sorption of water into the material and not favourable to the growth fungi [19]**.**

Conclusions

The oil heat treatment process increases the densities of the treated *A. mangium*. The densities increase from the bottom to the top portion of the tree. The oil-heat treatment increased the durability of *A. mangium* wood.

The attack of *G. trabeum* reduced from 0.54%-4.55%at top, middle and bottom, respectively. The *C. versicolor* attacks reduced from 4.78%-15.20%, 3.10%-12.69%, and 1.87%-10.19% at the top, middle and bottom portions, while the *P. sanguineus* attacks from 3.71%-10.18%, 5.74%-14.59%, and 4.37%-17.08% at the bottom, middle, and top portions.

For effective treatment, it is recommended to apply a temperature of 200°C to 220°C and duration between 60 to 90 minutes for enhancing the durability of the *A. mangium* wood.

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