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Scanning Electron Microscopy Study of the Effectiveness Oil Heat Treatment on 10-years-old teak wood in ground contact test

[1,2*]Mohamad Saiful Sulaiman, [1,2]Razak Wahab, [1,2]Nasihah Mokhtar, [1]Taharah Edin
[1,2]Sofiyah Mohd Razali and [1,3]Ros Syazmini Mohd Ghani

[1]University of Technology Sarawak (UCTS), 96000 Sibu, Sarawak, Malaysia
[2]Centre of Excellence Wood Engineered Products, UCTS 96000 Sibu, Sarawak, Malaysia
[3]University of Tasmania, Launceston, TAS 7250, Australia

Abstract: The microphotograph structure properties were studied on the performance of the 10 years old *Tectona grandis* wood that has undergone 3 months in a ground contact test. The heat treatment process at selected temperatures of 160°C, 200°C, and 240°C was applied for 2 hours in a specially designed oil heat-treatment machine powered by electricity. The oil heat-treated wood, including the control samples, were buried 80% into the ground at a randomly selected site exposed to rain and sun conditions. The wood was cut into sizes of 0.5 cm x 1 cm x 10 cm, being buried in the ground. The distances between the samples are planted at 5 cm apart. At the end of the testing period, all wood samples were taken, clean and dried desiccators. Once ready, the samples were studied for changes in the anatomy and microstructures using Scanning Electron Microscope (SEM). In addition, changes were examined between the treated and untreated teak samples. The oil heat-treatment process alters teak wood's cell structure, especially those treated at extreme temperatures. Observations focused on the cell structure, particularly at the fibre and parenchyma.

Keywords: *Tectona grandis,* cell structure, fungi, oil curing method, cross-sectional.

INTRODUCTION

Teak (*Tectona grandis*) is widely distributed in tropical hardwood native to India, Sri Lanka, and Southeast Asia [1], and it is a leafy tree of the family Verbenaceas that reaches up to 30 m in height. Teak has a density between 650 and 750 kg/m^3, with an average of 690 kg/m^3 at 12% humidity. It is considered a heavy wood of medium hardness [2];[3]. Teak wood has a medium bending resistance, minimum unbending nature and impact resistance, high compression resistance, and a moderate degree of bending with steam [2]. This deciduous tree is considered a precious wood in wood-based industries. It is recognized as one of the most endurable wood favoured for furniture, especially for outdoor use, because it is known to be capable of withstanding rain, sun, frost, and snow. Even after years of exposure to these harsh elements, teak furniture can remain stable in its form though with slight changes in its colour but some consider the natural beauty of the colour change to be aesthetic [4]. Due to the high demand for teak wood, it is only natural that teak is one of the main species planted in timber plantations [5]. This species had been known to be grown on the plantation for more than a century. Teak contains natural oils that repel water [6] and silica, preventing warping, deforming, and rotting [7]. The wood is also known to have great flexibility, enabling it to withstand high pressure without cracking. Generally, though teak has high natural durability and high resistance towards decay and insect attacks, the sapwood portion of the wood is comparatively weak towards insect attacks and rotting due to fewer extractives or deciduous oil in this wood portion. Early formed heartwood is also less durable than the heartwood from mature trees. Consequently, plantation teak wood quality at young age differs from forest harvested old teak trees [8].

Nevertheless, it has high resistance towards insects and fungi attacks, but they are not entirely immune to the insect attacks and rots, especially at the sapwood part. To further improve the durability and change to the service life of teak wood towards insects and fungi, heat treatment proves to be an effective and ecologically friendly method. A few methods are used for the heat treatment process on the wood or non-wood, it is sometimes known as rectified or torrefied wood due to the mild pyrolysis causing chemical transformation on the wood chemical components [9].

Corresponding Author: Mohamad Saiful Sulaiman. University College of Technology Sarawak (UCTS),
Email: saiful.sulaiman@ucts.edu.my

It is generally to improve its durability by fungal degradation, lower the equilibrium moisture content, lessen moisture deformation, and increase resistance towards the weather. Another type of heat treatment comes in the form of oil curing. Though it is already prevalent for wood to be treated with oil in olden times, not much has been said about the wood treatment using heated oil as a medium for heat treatment. The first case of treating timber with heated oil dates similar to heat treatment back in 1918 is said to be invented by Alfred Dunhill to manufacture tobacco pipes. Later in Europe, oil treatment has been used for treating selected wood species [10];[11]. In Southeast Asia, the oil curing method had been used for a long time, recommended by Wahab *et al*. [12];[13];[14] to enhance the durability of bamboo and rattans. On the morphological side, Teak fibre has a medium wall thickness with an average length of 700–1400 µm. Fibre pits are mainly restricted to radial walls, simple to border minutely. Fibres of teak are either exclusively septate or a mixture of septate and non-septate fibres. The septate fibres are usually evenly distributed [15].

The heat treatment process can alter the treated wood's cell morphology, affecting the other properties of wood [16]. By knowing what kind of changes are caused by heat treatment at the cellular level, it is possible to explain how the resistance of the wood towards insect and decaying matter is correlated to the morphology of the components of the cell [16].

The current study investigated the impact in the 10 years old *Tectona grandis* wood before and after undergoing the oil heat treatment via the Scanning Electron Microscopy observations at different magnifications [17].

MATERIALS AND METHODS

Sample Preparation

Three logs of 10 years of *Tectona grandis* were select randomly from a forest plantation owned by Superwood Sdn. Bhd. at Ranau, Sabah. The middle portion of the *Tectona grandis* log was cut out according to ISO 4471:1982 [33] to be used for this research. The central portions of the trees were subsequently cut into 2 cm x 6 cm x 75 cm. A total of 24 samples from the teak tree were prepared. Samples were directly vacuumed in plastic packaging and subsequently transferred to the laboratory to be heat treated [18]. The research parameter tested the effectiveness of temperature at 160°C, 200°C, and 240°C with independent variables, namely the wood portion (middle part), cross-section portion (sapwood section), age of the tree (10 years old), and duration of heat treatment (2 hours). All of the samples were conducted in triplicate.

Heat Treatment Process

Heat treatment was conducted according to Wahab *et al.* [19];[20] using the oil curing method. The treatment was carried out using a prototype model of an electrical heat-treatment machine, and crude oil palm oil was used as the heating medium. The palm oil were initially heated up to a temperature of 80°C first. The teak wood samples were placed into a metal cage and then submerged within the heated oil container. The first batch of the wood were taken out once the temperature reaches 160°C. The second batch was at 200°C, and the third batch was at 240°C (2 hours of treatment duration were allowed at each stage). A control panel located at the side of the machine was used to control temperature and duration of treatment closely [21];[22];[23]. The residual oil on the samples' surface was wiped clean with a cloth to prevent oil absorbance into the wood.

Graveyard Test

Heat-treated wood samples, including the control samples, were buried 80% into the ground at a randomly selected site exposed to rain and sun conditions [12]. Samples were cut into sizes of 0.5 cm x 1 cm x 10 cm for burying [16]. Waterproof tags with labeling codes are appropriately attached to the samples before the samples are buried. The distances between the samples are planted at 5 cm apart. Decaying fallen leaves and branches are piled at the site - the area with samples bordered with red and white tape. The samples are buried there for 3 months. The changes were illustrated via SEM.

Scanning Electron Microscopy (SEM) Analysis

After the three months, the wood samples were removed from the ground, cleaned, and oven-dried. They were then prepared for the SEM analysis, following technique outlines by Sulaiman *et al.*, [24];[25]. The samples were cut into blocks of about 5 mm x 5 mm x 5 mm using a penknife to fit the SEM sample plate stage. After being cut into appropriate sizes, specimens were sliced at the surface using a sharp blade to ensure a smooth surface on the sample for the SEM illustrated. The specimens' surface was coated with an ultra-thin coating of gold using a sputter coater machine, EMITECH K550X. ZEISS EVO® MA 10 Scanning Electron Microscope. Technically, the spot on the vessels, parenchyma, fibre, and middle lamella was observed. Images of the sample surfaces are recorded and labeled accordingly. The magnitude and scale of the image are highlighted in every figure. The SEM images for non-heat treated and heat-treated specimens before and after the graveyard test were compared. Observations focalized on the parenchyma, vessels, fibre, and middle lamella of a cell.

RESULTS AND DISCUSSION

Vessel Observation after Heat Treatment Process

Scanning Electron Microscopy (SEM) used in this study clearly illustrated the microstructures of teak wood at high magnifications. The vessel is a significant structure that is more distinct from its larger cell structure. Therefore, they were used in analyzing the changes that take between the oil heat-treated and untreated woods. Typical (Figure 1a), stretched (Figure 1c), and collapsed microstructures (Figure 1d) of the vessels were observed clearly. The elongated shape (Figure 1c) of inter-vessel pits as the structure of the vessels stretched. The structure of the vessels viewed in cross-section whereby recognized to be out of the standard round or oblong shape can also be an indicator of stretched vessels though being applicable for individual vessels only. SEM images obtained from the heat-treated samples showed that the samples of heated treated teak of 200˚C and 240˚C collapse vessels in observed samples and stretched vessels. Vessels of wood treated at 160˚C did not show signs of collapse on vessels structure, but symptoms of stretched vessels are relatively common. Figures 1a, 1b, 1c, and 1d show the SEM images at the transverse view of vessels from 10 years old teak samples. The control sample indicated a cross-section vessel in normal condition for Figure 1(a). At the same time, Figure 1(b) illustrated the stretched vessels noted by elongated inter-vessel pits. Nevertheless, Figure 1(c) indicated a collapsed vessel that is also out of shape. Then Figure 1(d) highlighted that a collapsed vessel wall.

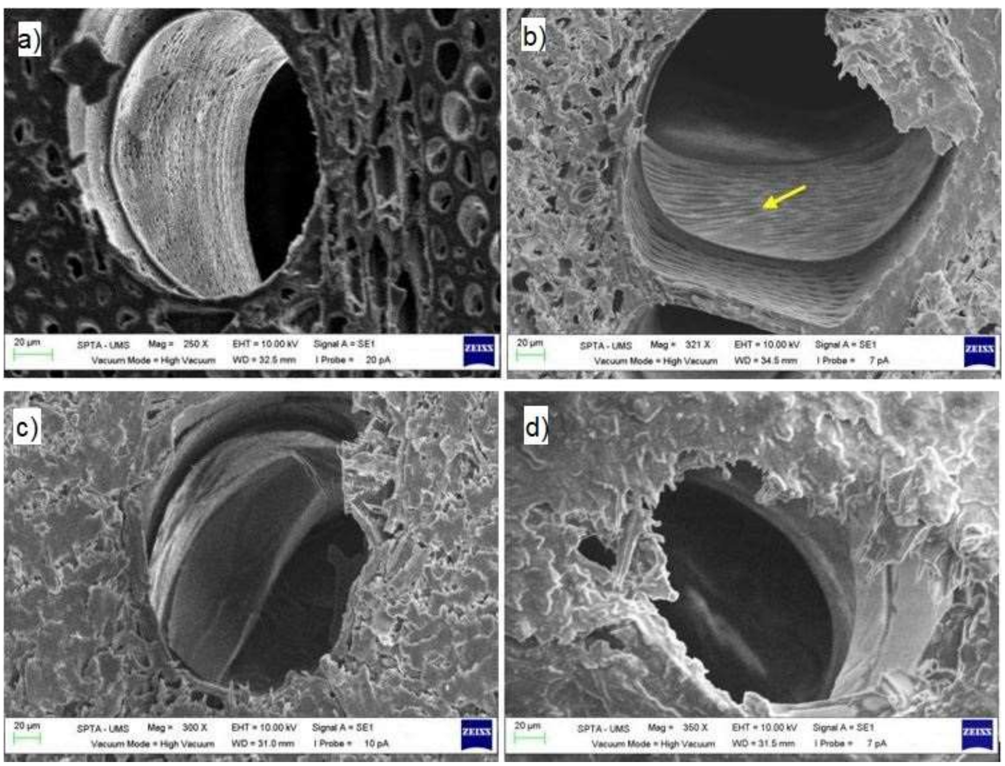

Figure 1: SEM images of transverse view vessels of 10 years old control samples and heat-treated samples at 160, 200, and 240˚C. a) Control sample. b) Heat-treated sample at 160˚C. c) Heat-treated sample at 200˚C. d) 260˚C heat treated.

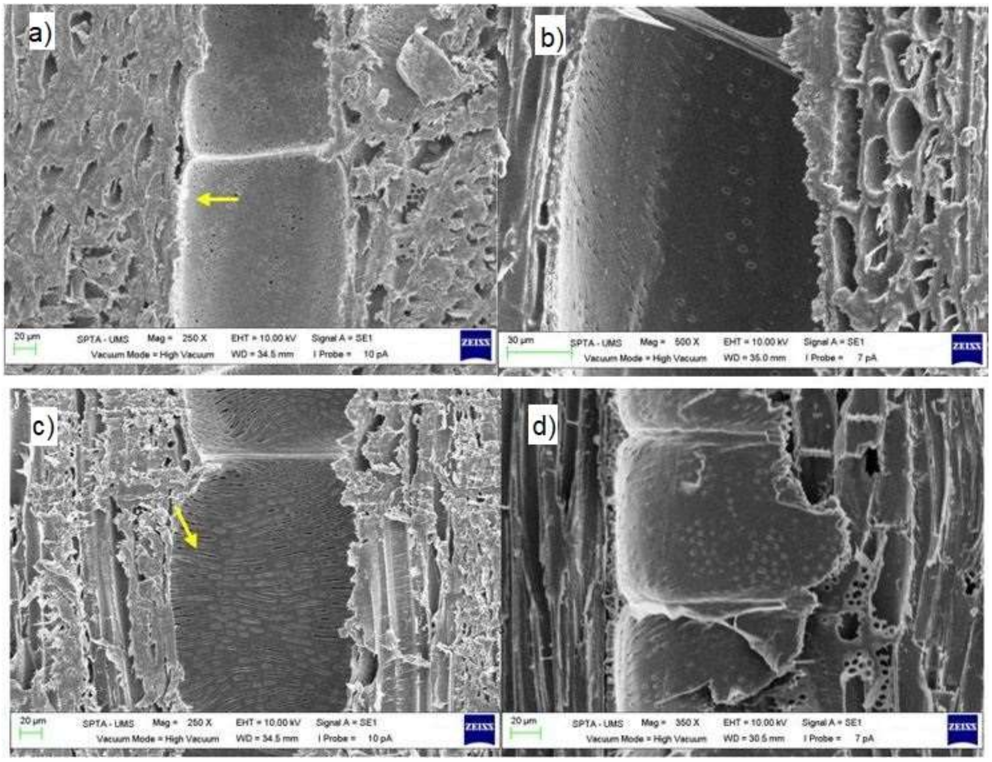

Figure 2: SEM images of a longitudinal view of vessels of 10 years old control samples and heat-treated samples at 160, 200, and 240˚C. a) Control sample. b) Heat-treated sample at 160˚C. c) Heat-treated sample at 200˚C. d) Heat-treated sample at 240˚.

Fiber Observation after Heat Treatment Process

SEM observations on fibre walls of the wood samples show differences in the microstructures between oil heat-treated and control untreated. It appears that the fibres of heat-treated samples tend to show fewer microfibrils on cut surfaces than control samples for both ages. However, the microfibrils' appearance could not be pinpointed since the microfibril strands of fibre cross-section could not be distinguished.

The SEM observations on the fibres at cross-section recorded that the threads of heat-treated samples showed deformation and even led to fracture at some samples. These were particularly observed in wood treated at extreme temperature of 240°C. Figure 3 highlighted that the fibres of heat-treated samples at a temperature of 160°C, 200°C, and 240°C of the wood samples shown signs of deformation on the shape of fibres at some parts. First, represented control samples featuring fibre in normal condition in Figure 3(a). Then the fiber is illustrated to deform in Figure 3(b). Furthermore, Figure 3(c) highlighted that fibers deformed and also fractured - next, samples showing irregular shapes or twisted fibres at Figure 3(d).

The heat treatment process affected the cell structure of the teak wood. Heat-treated samples had shown long vessels as well as collapsed vessels. This study demonstrated the heat treatment process that can cause the deformation of fibres at some parts of the wood. In addition, heat-treated teak wood also shown a decrease of microfibrillar angle in cell walls of fibres. When wood undergoes heat treatment process at high temperature, its chemical structure, especially at the cells wall is altered [26]. These changes affected the overall strength of these cell walls in particular and in the wood in general. Furthermore, applying the extreme temperature during the oil heat-treatment decreases the resilience of the cell walls causing deformation of the wood cells perceived, precisely vessel and fibre cells. The weakening of cells is due to the heat process causes the thermal degradation of the ultrastructure of the cell walls.

The degradation of hemicellulose occurred after the dehydration of the wood. The main chemical components of the wood cell walls, comprised of hemicellulose, cellulose, and lignin, undergo glassy transitions starting at temperatures ranging from 130°C to 190°C, depending upon the moisture available [26]. Decomposition of hemicellulose is reported in the range of 150°C to 280°C by some researchers [27]. Wood begins to degrade above room temperature thermally, but it is not losing molecules other than water until the active pyrolysis begins. The threshold temperature for active wood pyrolysis has found at 220˚C [28]. Thermal degradation of lignin also proceeds after dehydration of the wood, although the relationship to the wood thermal degradation is unclear. Glassy transitions of lignin are reported at temperatures ranging from 130°C to 190°C, again depending upon the moisture available [26]. Thermal started decomposition of lignin has been placed at 200˚C [29], whereas active pyrolysis of lignin is designated at the range of 220 ºC. On the other hand, lignin will soften when the wood is heat-treated.

As mentioned earlier, applying heat treatment alters the chemical structure of cell walls, which decreases the strength of the cell walls and causing deformation of the wood cells. As a result, the overall strength of the wood decreased. In addition, there are significant differences in the mechanical properties, especially bending strength, reduce in thermally modified wood [30].

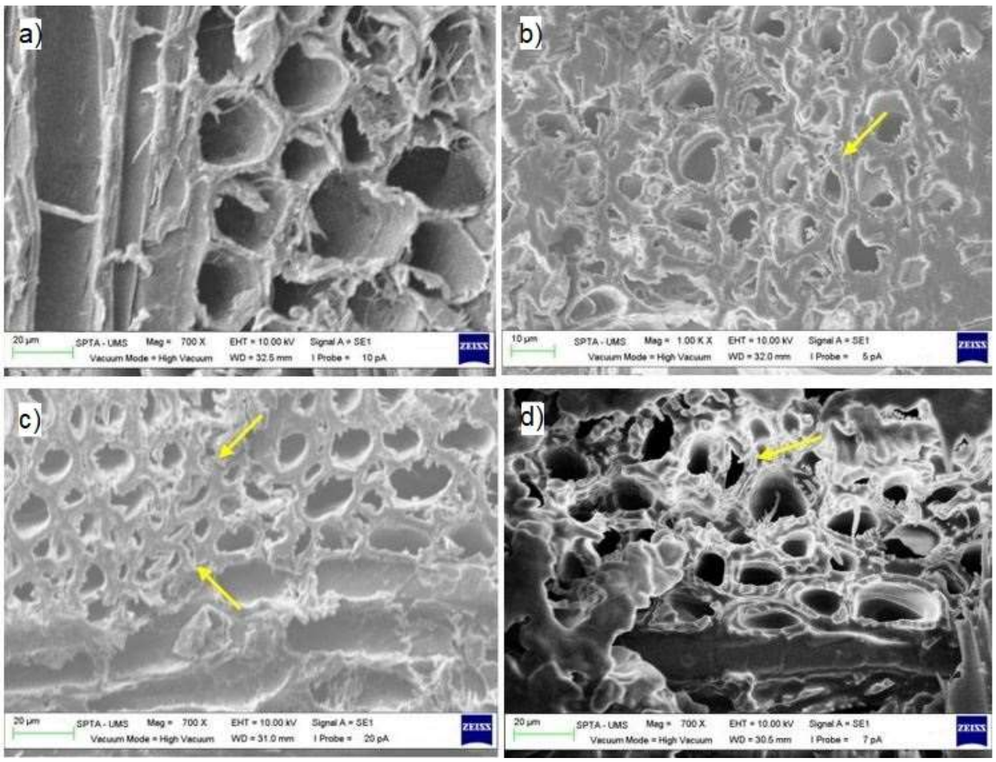

Figure 3: SEM images of a cross-section view of fibres of 10 years old samples. a) Control samples. b) Heat-treated samples at 160˚C. c) Heat-treated samples at 200˚C. d) 240˚C heat-treated samples.

SEM Observation on Parenchyma

SEM analysis of the heat-treated samples did reveal any changes or any damage to ray parenchyma cells (see Figure 1c and 1 d). However, the SEM observations do not show much difference in the axial parenchyma after heat treatment.

Changes Observed in Heat Treated Teak after Graveyard Test

After three months of graveyard test, SEM images obtained from the samples showed various indications of fungal attacks on control samples. For heat-treated samples at 160˚C, the image spotted a vessel at the inner lumen of warty walls. Besides that, hyphae had also been spotted at the lumen through their presence is very rare. However, heat-treated samples of 200˚C did not show signs of aggression by hyphae at vessel lumens. Also, heat-treated samples at 240˚C did not represent any attack signs by wood-decaying agents in the observed instances after graveyard testing. Figure 4 shows SEM images of cross-section vessel samples after the graveyard test. Figure 4(a) represented as a control sample highlighted hyphae intrusion in the vessel. In contrast, Figures 4(b) and 4(d) showed no signs of attack by fungi. Then, Figure 4(c) illustrated that vessels were collapsed and warty surfaces as the lumen cell wall.

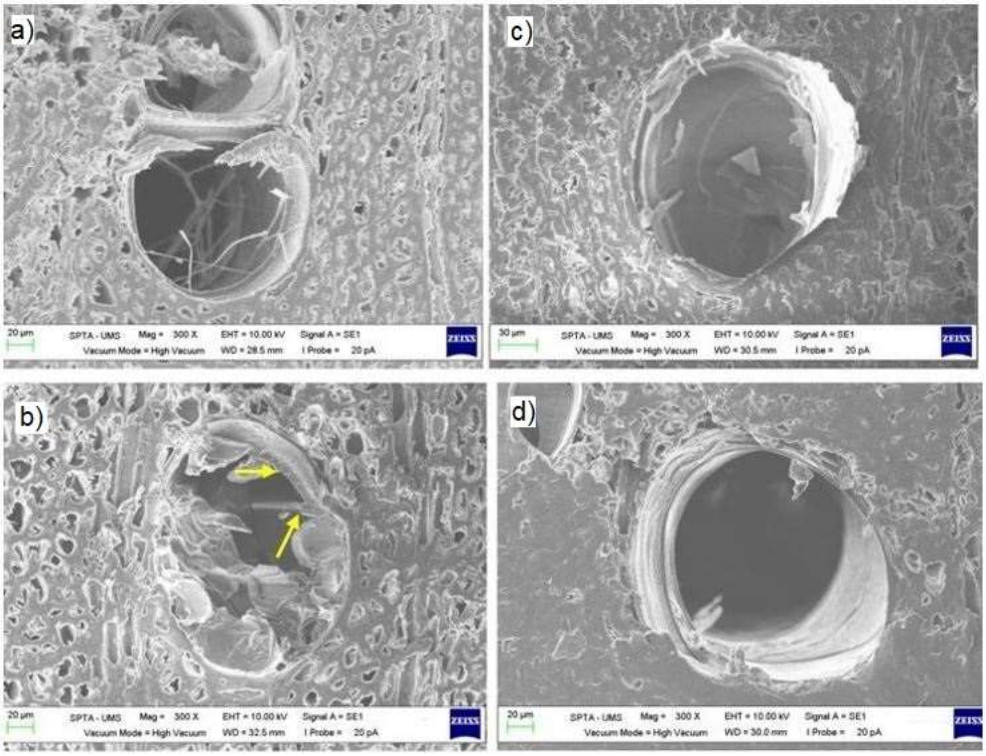

Figure 4: SEM images of 10 years old teak after graveyard testing showing cross-section of vessels. a) Control sample. b) 160˚C heat-treated sample. c) 200˚C heat-treated sample. d) 240˚C heat-treated sample.

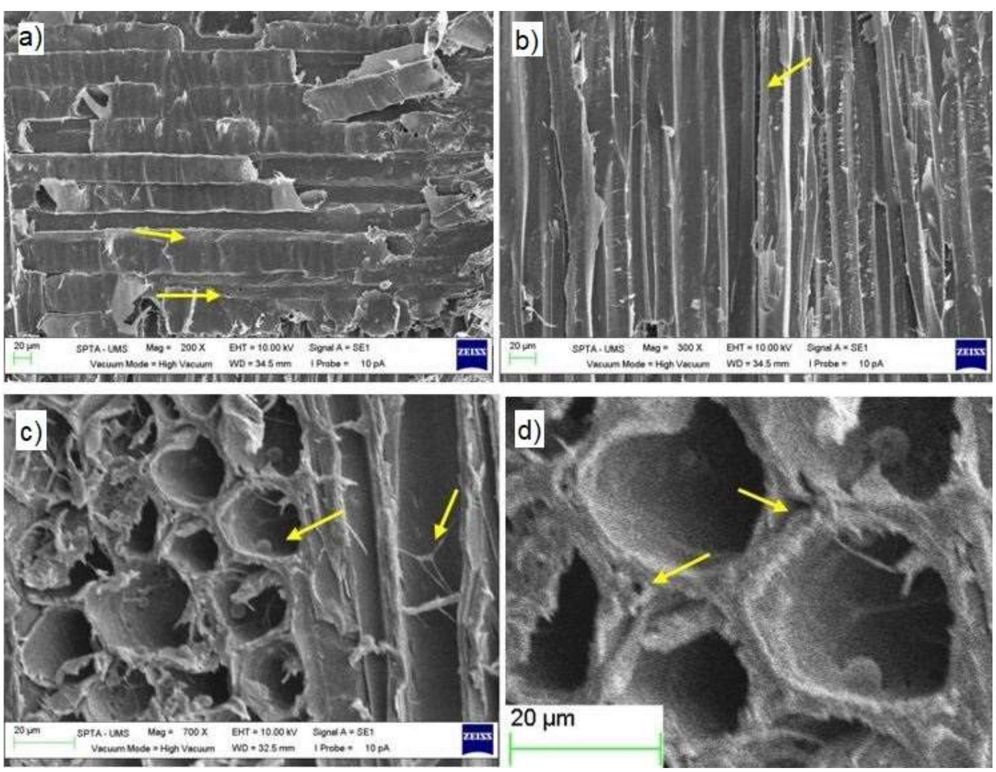

Figure 5: SEM images of 10 years old control samples after graveyard testing showing signs of attack by wood degrading agents. a) A radial view of ray parenchyma. b) Longitudinal fibres. c) Transverse view of fibres. d) Close-up view of the middle lamella of fibres.

The appearance of boreholes and hyphae signify fungi' attacks on the control untreated teak wood. Boreholes had been spotted quite commonly on ray parenchyma cells. Hyphae had also been recorded to appear at ray parenchyma cells. Walls between fibres also show separation or splits. Research on the control samples' fibres showed signs of attack by fungi since spore settlement of fungi can be seen. Figure 5 shows SEM images of control samples with an attack by fungi. Represented Figure 5(a) as a radial view of ray parenchyma showing boreholes. Then Figure 5(b) showed signs of separation between adjacent fibre cells. The fibres illustrated an attack by fungi and ray parenchyma showing hyphae filaments in Figure 5(c). Figure 5(d) showed an up-close view of the middle lamella between the transverse views of fibres. The middle lamella highlighted signs of being degraded and separated. Figure 6 spotted boreholes on ray parenchyma and axial parenchyma cells near the resin canal for control samples. Also, it highlighted that the degradation of the middle lamella on the fibre cellulose wall. Figure 6(a) represented boreholes, and Figure 6(b) showed signs of being degraded.

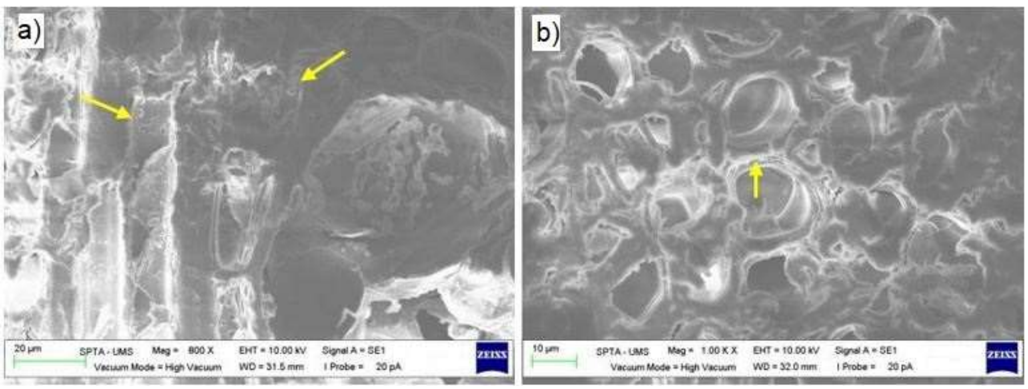

Figure 6: Control samples of 10 years old teak. a) Transverse view of ray parenchyma and axial parenchyma. b) Middle lamella of fibres.

Heat treatment enhances the durability of teak wood, especially against decaying fungi. Heat-treated teak had attested lesser or no fungi attacks signified by more minor boreholes and hyphae intrusion. According to Feist and Sell [31], the heat treatment directly affects the chemical constituents that believe to be less hygroscopic than untreated wood. The equilibrium moisture content of thermally modified wood becomes three to four times lower than that of untreated wood [32]. The moisture content of the wood and relative humidity and temperature of the environment are the main factors influencing fungi growth on wood. Heat treatment of wood decreases fungi formation because heat-treated wood becomes less hygroscopic. That means the amount of moisture content in the samples exposed to graveyard testing is higher in control samples than in heat-treated samples. The higher temperature of heat treatment applied, the equilibrium moisture content in wood would be lower [30]. Found that the heat-treated samples of 240˚C are scarce susceptible samples towards hyphae intrusion.

CONCLUSION

The study focuses on the effect of the oil heat-treatment process on the cell structure of the teak wood via the graveyard test and examines it by scanning electron microscopy analysis. The heat treatment process on the wood has a significant impact in enhancing their durability against wood-decaying fungi. Wood treated at 200°C is recommended and effective as it became most durable from attack by fungi after three months under the soil and did not alter much of the wood's microstructure. However, the heat treatment at a higher temperature alters teak wood's cell structure, leading to reduced strength in the cell walls. It explains the diminished mechanical properties of heat-treated wood proven in various heat treatment considerations. This finding also links to the increased dimensional stability of heat-treated. The primary purpose of heat treatment is to alter the wood cells' hygroscopic properties, leading to all of the positive effects of heat treatment.

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