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Role of Extractives in the Durability of *Neobalanocarpus heimii* and *Shorea falcifera* Heartwoods

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Wood durability refers to the resistance of the wood from decay agents such as fungi, termites, marine borers and weathering. Extractives isolated from the heartwood of durable hardwood species and some other plants species may provide alternatives to pest control because of their bioactive compounds. Wood extractives are known to affect wood resistance against fungi and termites attack. This study was carried out to determine the role of extractives of two natural durable woods namely *Neobalanocarpus heimii* (chengal) and *Shorea falcifera* (balau kuning) towards fungal decay and termites attack. A non-durable wood, *Dyera polyphylla* (jelutong paya), was used as control. Sequential Soxhlet extractions were done by using firstly mixture of 95% ethanol and toulene at a ratio of 2.32:1 (v/v), followed by 95% ethanol, 95% methanol and finally hot distilled water. The extracted and unextracted wood cubes were tested for decay resistance based on soil blocks decay test using white-rot (*Trametes versicolor*) and brown-rot (*Coniophora puteana*) decay fungi. A no-choice termite resistance test was carried out using a subterranean termite *Nasutitermes* sp. The weight loss due to fungal decay and termite attacks were determined after twelve weeks and three weeks of exposure, respectively. Chemically extracted test blocks were compared to unextracted blocks. Extracted durable species were also compared to non-durable controls. Sequential Soxhlet showed that *Shorea falcifera* yielded the highest amount of extractives at 19.81%. Results showed that extracted and unextracted blocks of *Dyera polyphylla a* non-durable wood recorded the highest weight loss in both fungi and termite attacks. Decay resistance test also showed that all extracted *N. heimii* and *S. falcifera* blocks exhibited significantly higher weight loss due to fungi and termites attacked compared to unextracted blocks. Overall results of these tests indicated that extractive content is primarily responsible for durability of *N. heimii* and *S.falcifera*.

Keywords: natural durability; heartwood; extractive free; soil block test; termite resistance test.

I. INTRODUCTION

Neobalanocarpus heimii (cengal) and *Shorea falcifera* (balau kuning) are among the Malaysian heavy hardwoods from the dipterocarpaceae family. Cengal also known as ‘Penak’ in Peninsular Malaysia is widely distributed in Selangor, Negeri Sembilan, Western Pahang and Southern Pattani in Thailand. It has been reported to occur in the state of Perlis and Melaka, and in diverse localities, on low-lying flat land as well as on hills up to 900 meter (Symington *et al.*, 2004). The cengal wood have air dry density ranging from over 915 to 980 kg/m³ that is very durable even under adverse condition (Yamamoto and Hong, 1988). Cengal is resistant to termite and fungi, and is among the strongest timbers in the world, that it is 50% stronger than teak (Symington *et al.*, 2004). It is commercially used for heavy construction, bridges, boat building, and also source for dammar penak, one of the finest natural dammars. The dammar penak has been used in the manufacture of varnishes (Orwa *et al.*, 2009). The

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heartwoods colour of cengal is light yellow-brown and known to have a high degree of decay resistance by virtue of its high extractive content (Kim *et al.*, 2006). According to Ahmad *et al.*, (2013), cengal heartwood contains high amounts of lignin and extractives. The reaction products of nitrobenzene oxidation showed that the lignin in cengal heartwood has more condensed structure and more guaiacyl lignin than that in the hardwoods growing in temperate zones. An aqueous wood extractives have been known to contain mainly polyphenols which have fungal inhibitory properties to *C. versicolor* (Yamamoto and Hong, 1988).

Shorea falcifera species is found in Peninsular Malaysia, Borneo and Sumatra. In Peninsular Malaysia, the local name of *Shorea falcifera* is Balau Kuning, while in Sarawak it is known as Selangan Batu Kering. Its heartwood colour varies from light red-brown to dark red. Balau timbers are used for all forms of the heavy construction, wharves, bridges, railway sleepers, boat building, power-line poles, heavy duty flooring, door and window frames, parquet flooring, joists, beam, rafters, and heavy duty furniture (Menon *et al.*, 2004).

The high durable tropical hardwood species usually contain high levels of extractives and provide durability, good dimensional and stability, and have been used extensively as construction materials (Schultz and Nicholas, 2002). Durable woods become more susceptible to the attack from bio-deteriorating agents after the heartwood extractives have been removed (Nagadesi and Arya, 2016). The presence of extractives can also lower the equilibrium moisture content, reduce swelling and shrinking of the wood by occupying the space where hygroscopic water could enter. It had also been proven that fiber saturation point can increase after the extractives are removed from the wood. The durability of tropical hardwood is attributed to the toxicity and water-repellence of extractives composition (Xie *et al.*, 2012). The extractives isolated from natural resistant heartwood and some other plants species may provide alternatives in pest control because they possess bioactive chemicals (Tascioglu *et al.*, 2012).

Wood extractives are non-structural components of wood that typically concentrated in the heartwood and are often produced by the standing tree as defensive compounds to environmental stresses (Kirker *et al.*, 2013). The natural compounds such as monoterpenes, amines, flavonoids, coumarins, terpenoids, phenolics polyketides, alkaloids, and phenylpropanoids from different plants comprise many organic compounds that have biofungicidal properties that can inhibit the growth of fungi. Several synergistic wood extracts combinations also been evaluated against wood decay, such as tannineborate combinations, condensed tannins from bark complexed with copper, and combinations of heartwood extractives and quaternary ammonium compounds (Hwang *et al.*, 2007; Thevenon *et al.*, 2009). Therefore, this study aims to determine the role of extractives in durability of *N. heimii* (cengal) and *S. falcifera* (balau kuning) by removing as much extractives as possible from the test blocks. The evaluation was carried out by measuring extracted and unextracted test blocks deterioration due to decay fungi and termites.

II. METHODOLOGY

A. Preparation of Wood Sample Blocks

The mature heartwood samples of *N. heimii* and *S. falcifera* were collected from Raub Sawmill Sdn. Bhd. Pahang. Quarter sawn planks were ripped to obtain wood blocks of size 1 cm^3 cubes. *Dyera polyphylla* (jelutong paya), a non-durable species was used as a control. Forty-six defect-free wood blocks from each species of *N. heimii*, *S. falcifera* and *D. polyphylla* were randomly selected and weighed. Subsequently the wood blocks underwent oven drying at 105°C until constant weight was achieved and recorded as oven-dried weight.

B. Extraction of Wood Block

Twenty-three blocks from each species were randomly selected for extractions. The extraction was carried out using Soxhlet method, according to the ASTM D1105-96 (2002) and Kirker *et al.* (2013), with minor modifications. The first extraction was done in 250 ml of mixture of 95% ethanol and toluene at a ratio of 2.32:1 (v/v) for 10 hours. Then, the blocks were rinsed with 95% ethanol and continued to be extracted in 250 ml of 95% ethanol for another 10 hours. After rinsing with methanol, extraction was continued again in 250 ml of methanol for another 10 hours. After that, the final extraction was done in hot distilled water for 10 hours. The extracted blocks were then oven-dried at 105 °C until constant weight and the weight were recorded.

C. *Soil Block Test*

The test fungi used for the soil block test were *Trametes versicolor* (white rot fungi) and *Coniophora puteana* (brown rot fungi). The fungi were cultured in 2% (w/v) of Malt Extract Agar (MEA) at room temperature for a week. The process of culturing was observed from time to time to prevent any contamination, and if contamination was detected, new inoculation process would be conducted again. The decay tests were done in sterilized soil culture plastics bags containing 250g of top soils together with the wood block, filter paper and plastic wire mesh. One sample wood block of *N. heimii* and *S. falcifera* and *D. polyphylla* were placed in each plastics bags.

Ten extracted and unextracted blocks were exposed to each fungi. Filter papers were dipped in Malt Extract Agar (MEA) and placed in each plastic bag to provide satisfactory growth of the fungi. Then, they were incubated at room temperature for 12 weeks and were maintained in the dark condition. After the exposure and incubation, the blocks were cleaned from the mycelium and they were conditioned at 105°C until constant weight. Then, the weight losses due to decay fungi were determined.

D. *Termite Resistance Test*

Termite resistance test was conducted according to the American Society for Testing and Materials (ASTM) Standard D3345-74 (reapproved 1999) (ASTM, 1999) with minor modifications. Beatson jars were used and filled with river sand. In each container, the sand were filled up to one-third full, and then autoclaved at 121°C for 2 hours. Subterranean termite, *Nasutitermes* sp. was collected from deadwood found in UNIMAS arboretum. The *Nasutitermes* sp. termites comprise of 45 workers and five soldiers were then introduced into each of the containers. The test was conducted in the closed cabinet to provide dark condition for the termite, at the room temperature for three weeks. After three weeks, the blocks were then being cleaned up and conditioned in the oven at 105°C until constant weight. Wood blocks were visually assessed and weight losses were determined.

E. *Calculation*

a) Amount of extractives content :

$$\textit{Amount of extractives (\%)} = \left(\frac{W1-W2}{W1}\right) x\ 100 \quad (1)$$

where, W_1 = Mass of the wood blocks before extraction
W_2 = Mass of the wood blocks after extraction

b) Determination of weight loss due to wood decay fungi :

$$\textit{Weight Loss (\%)} = \left(\frac{Wa-Wb}{Wa}\right) x\ 100 \quad (2)$$

where, W_a = Mass of conditioned wood blocks before exposure to fungi
W_b = Mass of conditioned wood blocks after exposure to fungi

c) Determination of weight loss due to termite attack:

$$\textit{Weight loss (\%)} = \left(\frac{X1-X2}{X1}\right) x\ 100 \quad (3)$$

where, X_1 = Mass of conditioned wood blocks before exposure to termite
X_2 = Mass of conditioned wood blocks after exposure to termite

d) Number of dead termites was recorded at the end of the test based on the following calculations:

$$\textit{Mortality rate of termites (\%)} = \left(\frac{N2}{N1}\right) x\ 100 \quad (4)$$

where, N_1 = Number of initial termites
N_2 = Number of dead term

F. Data Analysis

One-way analysis of variance (ANOVA) was carried out to determine the differences in weight losses between extracted and unextracted wood blocks, and wood species. Further analysis of mean comparison was done using Turkey HSD test. All analyses were done using SPSS20.0.

III. RESULTS

Table 1 shows that the extractive content of *S. falcifera* was the highest (19.81%), followed by *N. heimii* (5.59%) and *D. polyphylla* (4.56%). The fact that *S. falcifera* heartwood colour is darker than *N. heimi* suggests that *S. falcifera* had significantly more extractives than *N. heimii* and *D. polyphylla*.

Table 1. Mean extractive content of wood blocks of *N. heimii, S. falcifera and D. polyphylla*.

Wood species	**Extractive content (%)**
Neobalanocarpus heimii	5.59a*
Shorea falcifera	19.81b
Dyera polyphylla	4.56a

*Means within the column followed by the same letter are not significantly different at 5% level.

The classifications of wood resistance towards *Trametes versicolor* (TV) and *Coniophora puteana* (CP) based on the ASTM D 2017-05 (2005) showed that the extracted and unextracted *N. heimii* heartwoods are categorized as highly resistance. Based on the ASTM D 2017-05 standard, the weight losses of 0-10% in soil block is considered as highly resistance. The differences in weight losses due to TV and CP between extracted and unextracted wood blocks are shown in Figure 1 and Figure 2, respectively. The weight loss of extracted wood blocks of *N. heimii* and *S. falcifera* were significantly higher than compared of unextracted wood blocks. *Shorea falcifera* performed differently than *N. hemii* and less durable after extraction when exposed to CP but maintained its high resistance to TV. Our findings indicated that *D. polyphylla* was not resistance to decay fungi.

Figure 1. Mean weight loss of *Neobalanocarpus heimii, Shorea falcifera and Dyera polyphylla* after 12 weeks exposure to *Trametes versicolor*.

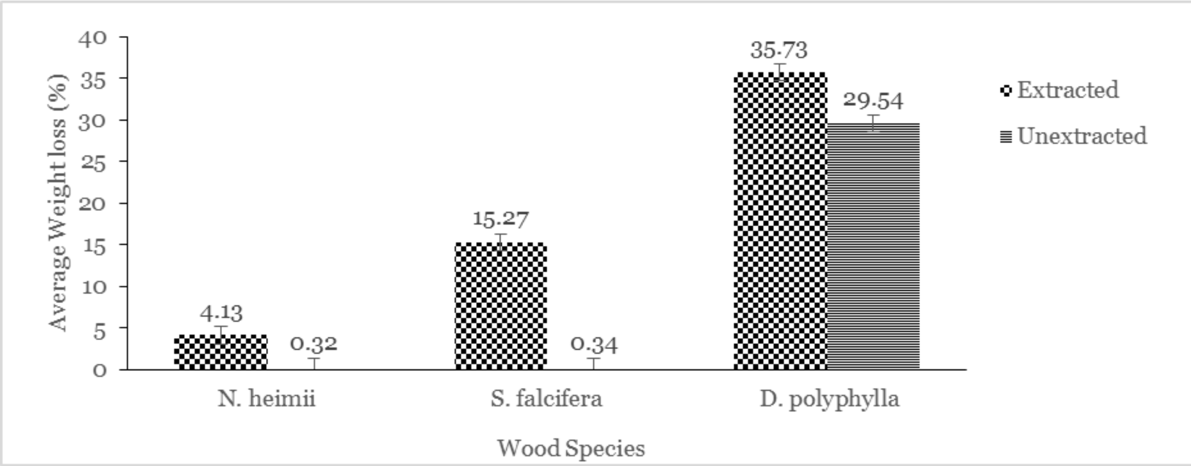

Figure 2. Mean weight loss of *Neobalanocarpus heimii, Shorea falcifera and Dyera polyphylla* after 12 weeks exposure to *Coniophora puteana*.

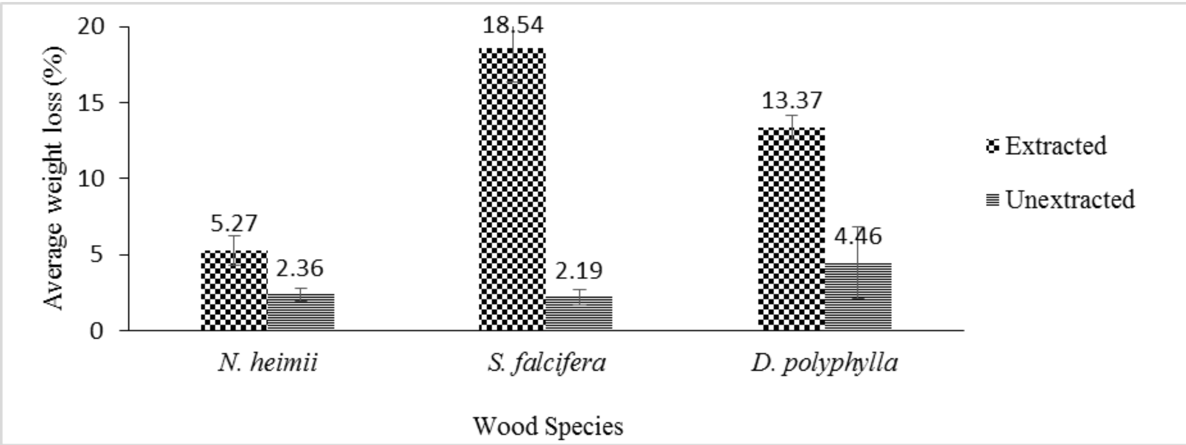

Figure 3. Mean weight loss of *Neobalanocarpus heimii, Shorea falcifera* and *Dyera polyphylla* after three weeks exposure to the *Nasutitermes* sp. termites.

Table 2. Mean visual ratings of *Neobalanocarpus heimii, Shorea falcifera*, and *Dyera polyphylla* after exposed to 45 workers and 5 soldiers of *Nasutitermes* sp. for three weeks in a no-choice laboratory test.

Wood species	Visual Ratings	
	Extracted	Unextracted
Neobalanocarpus heimii	8.33	8.66
Shorea falcifera	2.00	8.00
Dyera polyphylla	4.00	5.00

Table 3. The mortality rate of termite after three weeks exposure to the 45 workers and 5 soldiers of *Nasutitermes* sp. termite.

Wood species	Sample block	Mortality (%)
Neobalanocarpus heimii	Extracted Unextracted	100 100
Shorea falcifera	Extracted Unextracted	100 100
Dyera polyphylla	Extracted Unextracted	98 98

The weight loss of *D. polyphylla* was also observed to be significantly high. This indicates that the extracted blocks of *N. heimii*, *S. falcifera* and *D. polyphylla* were greatly consumed by termites as compared to the unextracted blocks from these three species of trees.

The average mortality of termite after three weeks of exposure to wood blocks is shown in Table 3. Both *N. heimii* and *S. falcifera* resulted 100% mortality of *Nasutitermes* sp. termite. The control wood, *D. polyphylla* resulted in 98% of mortality rate.

IV. DISCUSSION

The wood which recorded the weight loss from 10 % to 30 %, are classified as non-durable in any laboratory condition test (Findlay, 1985). The classification of wood resistance towards fungi tested, *Trametes versicolor* (TV), according to Findlay (1985), showed that the unextracted of *N. heimii* (2.30%) and *S. falcifera* (0.17%) heartwoods due to the average weight loss categorized in class 1, very durable timber. The unextracted *D. polyphylla* (39.66%) heartwoods is categorized in class v, perishable timber. While the wood resistance towards fungi tested, *Coniophora puteana* (CP), according to Findlay (1985), showed that the unextracted of *N. heimii* (0.32%) and *S. falcifera* (0.34%) heartwoods due to the average weight loss categorized in class I, that is very durable timber. While extracted *D. polyphylla* (29.54%) heartwoods is categorized in class IV, non-durable timber. Based on this classification, *D. polyphylla* are classified as non-durable and it is the most susceptible wood against decay test and termite attack. The other two wood blocks which were *N. heimii* and *S. falcifera* are classified under the durable wood species. The results showed that the extracted wood were more susceptible toward TV and CP decay fungi and suggest that *N. heimii* and *S. falcifera* heartwoods extractives had antifungal properties. Mohebby (2003) stated that, the white rot fungi is more dangerous than the brown rot fungi as it can degrade all the content of cell wall, which are cellulose, hemicellulose and lignin, thus causing greater damage and weight loss to the wood. White rot fungi and brown rot fungi possess some free radical components to disrupt the cell wall of the wood, so that they could increase the pore size to penetrate their enzymes into the cell wall for consumption (Flournoy et al., 1993).

According to the Yamamoto and Hong (1989), in his study of location of extractives and decay resistance in some Malaysian hardwood species, shows that in general of 24 selected timber species, Chengal (*Neobalanocarpus heimii*), Balau (*Shorea* sp.), Giam (*Hopea* sp.), and Rengas (ANACARDIACEAE), contain more extractives than the non-durable group which are Perupok (*Lophopetalum* sp.), Jelutong (*Dyera costulata*), Ramin (*Gonystylus* sp.), and Rubberwood (*Hevea brasiliensis*). Methanol extractives have been shown to contribute to the decay resistance of chengal (Yamamoto & Hong, 1988). The chengal heartwood contains high amounts of lignin, guaiacyl lignin and extractives (Kim et al., 2006). According to Wong et al. (2005), chengal wood (31.4%) contains similarly high amounts of lignin compared to belian wood (29%). According to Yamamoto and Hong (1988), Jelutong that non-durable species did not have extraneous materials in the lumina of any cell types. Unextracted and methanol extracted blocks of Jelutong had similar decay features where the ray and axial parenchyma cells were more selectively decayed than fibres. The amount of extractives sometimes indicates the degree of wood decay resistance (Takahashi & Kishima 1973). Another way to recognize the natural durability of the wood species is by looking at the colour of the heartwood. Wood colour is related to extractives content (Gierlinger *et al.*, 2004). The high natural durability wood species is darker in colour compared to the less natural durability wood species and this often taking as the indicator to test the natural durability (Wong, 2002).

The types of extractives in the timber species have a different function that influences the durability of wood (Gold *et al.*, 1999). The results in the soil block test were in agreement with Kirker *et al.* (2013) studies, where they observed that extracted *Robinia pseudoacacia* (black locust), *Prosopis glandulosa* (honey mesquite), *Paulownia tomentosa* (paulownia) *Catalpa* sp. (catalpa) were significantly decayed by *Trametes versicolor*, *Gloeophyllum trabeum* and *Postia placenta*. In some cases, extractive show activities that are specific to the organism that usually attack the timber. The weight loss differences of wood blocks can be influenced by several factors such as the natural durability of the wood, the fertility of the soil, adaptability (Nilsson *et al.*, 1992), climatic and geographic region (Zabel and Morrell, 1992), decay rate of fungi (Anangnost and Smith, 1997), and genetic factors (Campbell and Clark, 1960).

The extracted wood blocks shows significantly higher weight loss as compared to unextracted wood blocks after being exposed to *Nasutitermes* sp. termite can be correlated with the mortality rate of the termites. This can be related to the subterranean termites that are well-known as the most destructive and widely distributed species all over the world, causing severe damage to wood materials and cellulose materials that are unprotected (Lee *et al.,* 2007). According to Gold *et al.* (1999), the subterranean termites attack wood structure by building tunnel that connect their nests to the wood structure especially *Nasutitermes* sp. that was observed to make a few tunnels in the sand to build its nest. Bultman and Southwell (1976) studied the natural resistance of the wood of 114 arboreal species in the Panama forest and stated the wood density is one of the factor of wood resistance to subterranean termites. Wood density had the highest effect on the rate of degradation and which is the higher the wood density the more resistance toward termites (Shanbhag and Sundararaj, 2013; Bultman et al. 1979; Abreu and Silva, 2000). This is because the harder wood can reduce the chewing ability of termite resulting in lower weight loss (Watanabe et al., 2005). Ragon et al. (2008) conclude that the higher the cellulose content, the higher the susceptibility to termites attack. According to Shanbhag and Sundararaj (2013), the chemical constituent such as cellulose, lignin and total phenolic content of wood influenced the rate of degradation. The higher the resistance of wood species related with the higher of lignin and phenolic content. Cellulose is a primary food source for termites and acts as one of the factors that attract termites towards wood species, which explains the significant positive correlation of cellulose content and wood degradation (La Fage and Nutting, 1978). Lignin an indigestible to termites that acts as a physical barrier of woods (Walcott 1946; Syafii et al. 1988).

Wood extractives may play a role in inhibiting the termites attack. It was reported that subterranean termites has ability to detect the heartwood that contain extractives, anti-oxidant activity or toxicity and avoid them from degrading wood (Ragon *et al.*, 2008). According to Moore (1979), and Barbosa et al. (2003), chemical component properties in some wood species have high correlation between percentage of extracts toxicity and the resistancy of natural durability of woods to termites. Tsunoda (1990) concluded that the wood extracts compositions are valuable for better understanding of resistance to biodegradation. Furthermore, environmental moisture also plays an important role in termite attack. Some researchers also conclude that specific wood chemicals can initiate long-distance recruitment of termites to their food sources, given that the wood is preference food source for the termite colonies (Price, 1997; Waller & La Fage, 1987).

V. CONCLUSION

The findings from this study showed that the removal of extractives content can significantly affect the durability of *N. heimii* and *S. falcifera* following exposure to white rot and brown rot fungi. Extracted wood blocks were also found to be more susceptible to termites attack. However, extracted and unextracted *N. hemii* blocks were still considered as highly resistance when exposed to both decay fungi and termites. This study showed that the extractive contributed to the resistance of the two durable species. However, it is important to note that the extractives alone may not be the sole factor influencing to the decay resistance of *N. heimii* and *S. falcifera*.

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