EFFECTS OF EXTRACTIVES ON THE DYNAMIC WATER SWELLING BEHAVIOUR AND FUNGAL RESISTANCE OF MALAYSIAN HARDWOOD

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XIE Y, HILL CAS, SUN DY, JALALUDIN Z & WANG Q. 2012. Effects of extractives on the dynamic water swelling behaviour and fungal resistance of Malaysian hardwood. Effects of extractives on the dynamic water swelling properties and fungal resistance of Malaysian tropical hardwood species, namely, sesendok (*Endospermum malaccense*), acacia (*Acacia mangium*) and chengal (*Neobalanocarpus heimii*) were determined. They exhibited extractive contents of 3.2, 7.5 and 24.9% respectively. During the swelling test, an induction period occurred at the initial stage. The swelling kinetic behaviour was non-linear throughout the entire process, especially for sesendok. Chengal, which had the highest extractive content, exhibited greater equilibrium swelling but lower swelling rate. Removal of extractives apparently resulted in increase in both the equilibrium swelling and swelling rate presumably due to easier access of water to the wood cell wall. Throughout the 48-hour immersion period, chengal in the tangential direction exhibited double component swelling profile. After incubation with brown- and white-rot fungi, the extracted chengal exhibited considerably higher mass loss than the non-extracted samples. A distinct difference in the decay behaviour of non-extracted and extracted chengal might be attributed to hydrophobic characteristics and cell wall bulking effects imparted by the extractives.

Keywords: Tropical wood, cell wall bulking, swelling rate, wood decay

XIE Y, HILL CAS, SUN DY, JALALUDIN Z & WANG Q. 2012. Kesan hasil ekstraktif terhadap kelakuan pembengkakan air dinamik dan kerintangan terhadap kulat bagi kayu keras Malaysia. Kesan hasil ekstraktif terhadap ciri pembengkakan air dinamik dan kerintangan terhadap kulat pereput bagi spesies kayu keras tropika Malaysia iaitu sesendok (Endospermum malaccense), acacia (Acacia mangium) dan chengal (Neobalanocarpus hermii) dikaji. Kandungan hasil ekstraktif mereka masing-masing 3.2%, 7.5% dan 24.9%. Semasa ujian pembengkakan, tempoh aruhan berlaku pada peringkat awal. Kelakuan kinetik pembengkakan adalah tidak linear sepanjang proses, terutamanya untuk sesendok. Chengal yang mempunyai kandungan hasil ekstraktif yang paling tinggi menunjukkan pembengkakan keseimbangan yang lebih tinggi tetapi kadar pembengkakan yang lebih rendah. Penyingkiran hasil ekstraktif nampaknya mengakibatkan peningkatan dalam pembengkakan keseimbangan dan kadar pembengkakan. Ini mungkin disebabkan oleh kemudahan kemasukan air ke dalam dinding sel kayu. Sepanjang rendaman air selama 48 jam, profil pembengkakan chengal adalah dua kali ganda pada arah tangen. Selepas pengeraman dengan kulat pereput perang dan putih, chengal yang diekstrak menunjukkan kehilangan berat yang lebih tinggi berbanding sampel yang tidak diekstrak. Perbezaan yang ketara antara kelakuan pereputan chengal yang tidak diekstrak dengan yang diekstrak mungkin disebabkan oleh ciri hidrofobik dan kesan pengisian dinding sel yang ditunjukkan oleh hasil ekstraktif.

INTRODUCTION

As traditional building material, wood exhibits pleasant aesthetics and high specific strength. Therefore, it is being used extensively as structural and non-structural parts in buildings. However, dimensional instability (shrinking and swelling) due to variable environmental relative humidities can bring about and/or facilitate surface checking, cracking, cupping and warping, mould growth, fungal decay and strength loss, thereby reducing service life (Sandberg 2005, Blom & Bergström 2006). The highly durable tropical hardwood species normally contain high levels

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of extractives, display good dimensional stability and durability and have been used extensively as construction materials. The durability of tropical hardwood is attributed to the toxicity and water repellence of extractive compositions (Taylor et al. 2002, Nzokou & Kamdem 2004). However, with the increase in environmental awareness, demand for durable tropical wood has reduced. This has led to increasing interest in the use of different wood modification technologies applied to temperate plantation species in order to obtain properties comparable with durable tropical hardwood. Apart from enhanced durability, many wood modification methods result in improved dimensional stabilisation. Among these methods, chemical modification renders nondurable wood dimensionally stable by depositing a chemical in the wood cell wall (bulking effect) and/or by grafting onto or cross-linking with cell wall polymers (Hill 2006). For example, acetylated wood is more stable dimensionally and contributes to great improvements in weathering and coating performance, and resistance against fungi (Beckers et al. 1998, Evans et al. 2000, Hill et al. 2005). Wood modified with 1,3 dimethylol-4,5-dihydroxyethyleneurea (DMDHEU) can achieve anti-swelling efficiency of 60% at weight per cent gain level of 25%. DMDHEU-modified Scots pine wood was more resistant to decay fungi (Militz 1993, Yusuf 1996) and exhibited improved weathering and coating properties (Tomazic et al. 2004, Xie et al. 2005, Xie 2006, Xie et al. 2008). The mechanism of the above improvements is assumed to be cell wall bulking resulting from chemical deposition (Hill et al. 2005) in which the chemicals normally do not show any toxicity to decay fungi (Verma et al. 2005).

Compared with durable tropical wood, a common characteristic of chemically-modified wood is the cell wall bulking effect caused by the presence of extractives or chemicals. In order to ascertain whether chemicals deposited in the cell wall performed a similar function to in situ extractives in cell walls, the influence of extractives on the wood swelling behaviour was examined. The dynamic swelling profile of wood has previously been measured using real-time monitoring apparatus. The swelling behaviour of several North American species in different solvents was determined and the effects of temperature, density and extractives on the maximum swelling were studied (Mantanis et al. 1994a, b, 1995). They found that increasing the temperature and removing extractives greatly accelerated the swelling rate and resulted in increase in maximum swelling. Virta et al. (2006a) measured the swelling stress in the tangential direction of spruce (*Picea abies*) immersed in water. It was found that the viscoelastic and mechano-sorptive creep decreased swelling stress and the maximum swelling stress was about 1.2 MPa. Shukla and Kamdem (2009) measured water swelling profiles of nine tropical hardwood species from Cameroon using linear variable differential transformers. The tangential swelling rate was much higher than radial direction and there was no linear relationship between total swelling and wood density.

In this preliminary study, three Malaysian tropical hardwood (acacia, chengal and sesendok) which exhibited varying chemical compositions and extractive contents were used. Previously, Zaihan et al. (2009, 2010) studied the dynamic water vapour sorption behaviour of these three species. The water vapour sorption rate was found to be slowest for chengal and fastest for sesendok. Following on from here, the relationship between extractive content and the dynamic swelling behaviour was explored. The effects of extractives on the durability of chengal were also examined.

MATERIALS AND METHODS

Wood samples

Three tropical wood species, sesendok (Endospermum malaccense), acacia (Acacia mangium) and chengal (Neobalanocarpus heimii) were obtained from Malaysia. Sesendok and acacia are plantationgrown species that are mainly used to make match boxes, toys, furniture, plywood, charcoal and wood pellets (Mansor 1989, Rushdan et al. 2007). Due to its high extractive content, chengal wood is very durable to decay fungi such as Trametes versicolor (Yamamoto & Hong 1988). An investigation on the effect of extractives on the dimensional stability of this species could provide insights into how wood modification cell wall bulking methods could be employed. Chengal is used for heavy construction, in bridge making, railway sleepers, telegraph poles, boat building and sea defences. This species is under increasing pressure from loggers. If the role of extractives in imparting desirable properties, appropriate 'biomimetic' wood modification methods for alternative species could perhaps be developed. The wood species studied were all heartwood

and were carefully cut from one tree. Specimens measuring $20 \times 20 \times 200$ mm (radial × tangential × longitudinal) were prepared. Subsequently, wood blocks measuring $20 \times 20 \times 5$ mm (radial × tangential × longitudinal) were cut from these pieces to obtain a clear grain direction. Density and comparable year ring number were recorded. These blocks were oven dried at 105 °C for 24 hours and then allowed to cool to ambient temperature in a desiccator before weighing. For each wood species 20 replicates were prepared.

Extractive content and density determination

The different batches of wood blocks were soaked in flasks containing mixed solution of water and acetone (20:80 by volume). The flasks were stored in a water bath at 50 °C for 48 hours. Throughout extraction, the solution was changed every 12 hours. After extraction, the extracted wood blocks were taken out of the solution and carefully dried for 24 hours under controlled conditions of 20 °C and 65% relative humidity to avoid cracking as a result of rapid evaporation of solution. The oven-dry dimensions and weights of non-extracted and extracted wood blocks were measured after 24 hours of oven drying. The wood density was calculated as a value of oven-dry weight divided by oven-dry volume.

Dynamic swelling measurements

Prior to the swelling tests, wood samples were oven dried for 24 hours and then allowed to cool to ambient temperature in a desiccator. The wood block was quickly transferred from the desiccator into a flat-bottomed plastic container which was placed in an indicator with accuracy of 0.001 mm. The extension rod with a flat contact point had close contact with the sample which applied a relatively small force on its surface in the measuring direction. This prevented the wood block from floating up in water. The indicator was set to zero and distilled water (25 °C) was filled into the plastic container until the wood blocks were immersed. Measurement was done in an enclosed chamber at controlled temperature of 25 °C. The data were collected every 15 s using MeasurLink SPC REAL Time PLUS software. The swelling was calculated as the percentage of oven-dry dimension:

$$S = \frac{L_1 - L_0}{L_0} \times 100$$

where S is the swelling percentage, L_1 is the swollen dimension (mm) and L_0 is the ovendry dimension (mm). Three replicates were measured simultaneously on three indicators for 48 hours.

Brown- and white-rot testing

Basidiomycete decay testing was carried out according to modified EN 113 (EN 1996). The solvent-extracted and non-extracted chengal specimens were sterilised by gamma irradiation (2.5 Mrad) and then exposed to the fungi *Coniophara puteana* and *T. versicolor* for 12 weeks in jars covered with cotton wool. Additional sets of sterile controls were also placed in jars, but without fungal inoculation. For each fungus, 10 jars each accommodating one non-extracted and one extracted chengal sample were used. After incubation, the specimens were weighed and oven dried to determine the moisture content and mass loss.

RESULTS AND DISCUSSION

Density and extractive content

The wood used in this study exhibited densities from 0.33 to 0.77 g cm^{-3} (Table 1). The extraction process removed water- and solvent-borne extractives from the wood lumen, resin canal and cell wall. Thus, the wood density was reduced to different extents. Previous studies showed that chengal, acacia and sesendok had extractive amounts of 21.6, 8.1 and 1.5% respectively (Yamamoto & Hong 1988, Mohd Nor 1991, Zaihan et al. 2010). These values were comparable with 24.9, 7.5 and 3.2% obtained in this study (Table 1). Although chengal had high mass loss due to removal of extractives, it only shrunk slightly in the radial (0.5%) and tangential (1.1%) directions. The mass loss resulted in great decrease in density from the original of 0.77 to 0.58 g cm⁻³. This suggested that extractives were primarily located in the large cavities such as cell lumen and resin canal with only a relatively low proportion in the cell wall. Although 7.5% (by mass) extractives were removed from acacia, the extraction resulted in greater shrinkage compared with chengal, suggesting that those leachates were mainly located in the cell wall interior of acacia. The density of acacia was hardly affected by the

| Wood | Extractive content (%) | Dimensional change (%) | | Density (g cm ⁻³) | |
|----------|------------------------------|------------------------|-----------------|-------------------------------|---------------------|
| | | Radial | Tangential | Before extraction | After extraction |
| Sesendok | 3.17 (0.28) | -0.36 (0.22) | -0.80 (0.63) | 0.33 (0.05) | $0.32 \\ (0.04)$ |
| Acacia | 7.48 (0.41) | -1.29 (0.19) | -4.35 (0.92) | 0.61 (0.01) | 0.59 (0.01) |
| Chengal | 24.91 (1.15) | -0.47 (0.11) | -1.14 (0.18) | 0.77 (0.01) | 0.58 (0.01) |

 Table 1
 Effects of solvent extraction on extractive content, dimensional change and density of wood

Data in parentheses show standard deviations of 10 replicates

extraction process. There was little extractive content in sesendok and thus the extraction did not result in any apparent effect on the dimensions. The extraction process may damage and subsequently remove the wood structural composition such as lignin and hemicellulosic fragments, thereby, reducing the size of samples. However, the water/acetone mixed solution was reported as a relatively gentle extraction solvent (Mantanis et al. 1994b). The extracted samples after saturation by water could reach a maximum dimension comparable to that of the green nonextracted samples (not shown), suggesting that the extraction process did not substantially affect the cell wall structure.

Dynamic swelling behaviour

During the initial stage of water immersion, all wood samples (non-extracted and extracted) exhibited short induction periods (Figure 1). Higher extractive content in the wood resulted in longer induction period in both radial and tangential directions. The extracted wood also exhibited an induction period but shorter than the non-extracted ones. Induction periods were also observed in the swelling curves of wood (Mantanis et al. 1994b, Virta et al. 2006a, Shukla & Kamdem 2009). This is attributed to the lag time for the initial diffusion of solvent into the wood cell wall structure (Mantanis et al. 1994b) and the gradual increase in the number of cell wall capillaries along the fibre direction via the open pits and large parenchyma cells (West 1988, Virta et al. 2006b). In this study, chengal with an extractive content of 25% did not show any swelling within the initial 1 min. However, with decrease of extractive content, acacia (7.5%)

and sesendok (3.2%) had shorter induction period when they were immersed in water. This suggested that there was surface coverage of cell wall by extractives, which could slow down the access of water to cell wall polymers and could be responsible for the induction period. The swelling of wood, especially for sesendok with low density and extractive content, did not increase linearly in water within the initial 4 min (Figure 1). This is in contrast with that reported for several American species (Mantanis et al. 1994a).

Once water penetrates into the lumen, it can diffuse more easily into the cell wall, resulting in faster swelling rate than that in the initial induction period. The permeability of wood cell wall may significantly depend on the extractive content in the cell wall. The overall swelling profiles of wood samples up to 15 hours immersion (the total measuring time was 48 hours, but in order to exhibit more details at the initial stage, only the first 15 hours are shown) in both radial and tangential directions with time are presented in Figure 2. In the first nine hours of immersion, the radial swelling of non-extracted wood was negatively related to the extractive content. After that, sesendok and acacia samples reached an equilibration state, while chengal continued swelling to equilibrium value of 2.5% until approximately 35 hours (not shown in Figure 2a). Extraction did not influence the radial swelling profile of sesendok but apparently resulted in faster swelling and greater equilibrium swelling for acacia and chengal. This demonstrated that removal of extractives facilitated access of water to cell wall polymers.

By comparison with the radial swelling profile, the tangential swelling was greater for all wood

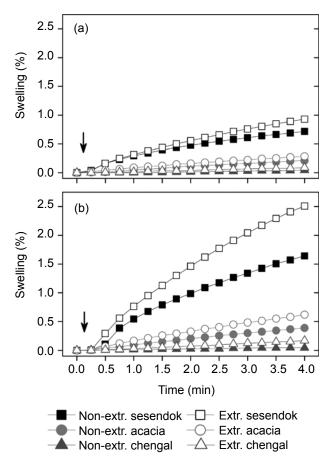


Figure 1 Initial swelling of non-extracted (Non-extr.) and extracted (Extr.) wood in (a) radial and (b) tangential directions within 4 min; arrows point to the induction periods

species (Figure 2b). Wood with higher extractive content swelled more slowly than those with lower extractive content (Figure 2b). As in the radial direction, swelling in the tangential direction did not reach equilibrium in the plotted time period. The equilibrium value was obtained after approximately 35 hours (not shown). After extraction, all three wood species exhibited faster and greater swelling along both tangential and radial directions through immersion. A more pronounced influence on tangential swelling could be observed. This may be explained by the denser cell wall substance, fewer pits and more extractives in the tangential cell walls (Hon & Shiraishi 2001). The swelling profile of non-extracted chengal apparently contained two curves, which had not been reported for other wood species (Figure 2b). The first curve exhibited a relatively slower swelling rate, which was probably due to the existence of extractives on the cell wall surface and in the lumen. The colour of the water gradually darken within three hours of immersion, showing that waterborne extractives were dissolved. This may result in an easier access of water to cell walls thereby speeding up the swelling. Loss of extractive material from the cell wall will also result in shrinkage competing with swelling due to ingress of water.

After 48-hour water immersion, all wood samples reached equilibrium. Equilibrium swelling tends to increase linearly with wood density (Mantanis et al. 1994a). The observation in this study, however, did not, exhibit such a tendency (Figure 3), which was consistent with the results presented by Shukla and Kamdem (2009). Non-extracted sesendok, acacia and chengal exhibited equilibrium swelling of 2–3% in the radial direction (Figure 3a) and 5-6%in the tangential direction (Figure 3b). The tangential swelling was a little lower than the value (8-9%) reported for spruce with density of 0.45 g cm⁻³ (Virta et al. 2006a). After extraction, the equilibrium swelling of all wood species increased. Chengal, which had greater density reduction due to removal of 25% extractives exhibited lower increase in equilibrium swelling than acacia which had extractive content of 7.5%(Figure 3). This further supports the suggestion

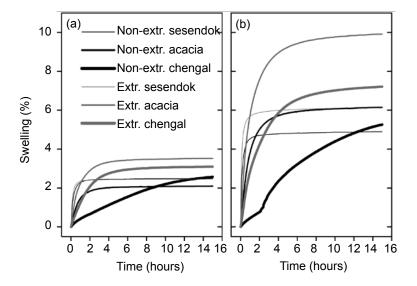


Figure 2 Dynamic swelling profiles of wood in (a) radial and (b) tangential directions before (Non-extr.) and after (Extr.) extraction

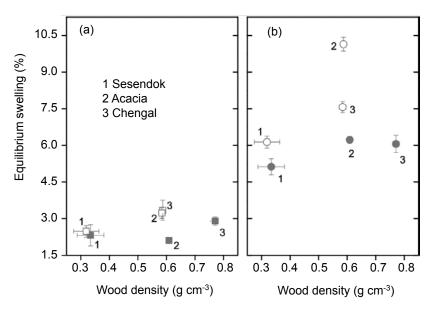


Figure 3 Equilibrium swelling of non-extracted (solid legend) and extracted (empty legend) wood in (a) radial and (b) tangential directions after 48-hour water immersion; error bars show standard deviations

that a considerable amount of extractives is located in the cell lumen of chengal and removal of them does not result in any substantial effect on the swelling capacity. This is because only the extractives deposited in the cell wall structure can substantially contribute to the changes in cell wall dimensions (Boiciuc 1970). Removal of extractives located in cell wall will perhaps result in a more hydrophilic characteristic. Thus, the cell wall can be accessed by water more easily and the space in the cell wall accommodating water is bigger (Mantanis et al. 1995).

Swelling rate of wood

In the initial induction period, the swelling rate of all wood samples exhibited rapid increase to maximum value (Figure 4). The swelling rate then decreased with immersion time until equilibrium state. The equilibrium state is determined by the balance of the swelling pressure of the cell-wallbound water and the increase in energy of the now stressed polymeric matrix of the cell wall (McClendon 1981).

The tangential swelling rates were higher than the radial. This is consistent with previous observations (Arevalo & Hernandez 2001, Chauhan & Aggarwal 2004). Sesendok swelled much faster than the denser acacia and chengal in both radial and tangential directions. After extraction, there was little increase in maximum swelling rate in both the radial and tangential directions for sesendok. However, a considerable increase was found for acacia and chengal, which demonstrated the strong effects of extractives on swelling rate. The increased swelling rates could be interpreted by the reduced surface energy due to removal of extractives, especially hydrophobic extractives such as waxes and long chain hydrocarbons (Maldas & Kamdem 1999). The other reason could be faster diffusion of water into the evacuated space in the cell wall where extractives were removed. The nonextracted sesendok, acacia and chengal required 2, 8, 35 hours to reach equilibrium swelling

(rate equals zero) respectively. However, they only needed 1.5, 6, 8 hours respectively after extraction (Figure 4).

Brown and white rot

The hyphae of both *C. puteana* and *T. versicolor* fully covered the non-extracted and extracted chengal samples (not shown), suggesting that the extractives in the wood were not toxic to these two types of fungi. After 12-week incubation, the non-extracted chengal samples inoculated with *C. puteana* and *T. versicolor* had moisture contents of 26 and 35% respectively, which were lower than 68 and 112% obtained on the extracted wood (Figure 5a). Higher moisture content could be attributed to easier access of water to the wood interior and higher activities of fungi due to removal of extractives. The non-extracted chengal did not exhibit any mass loss after incubation with both rot fungi. However,

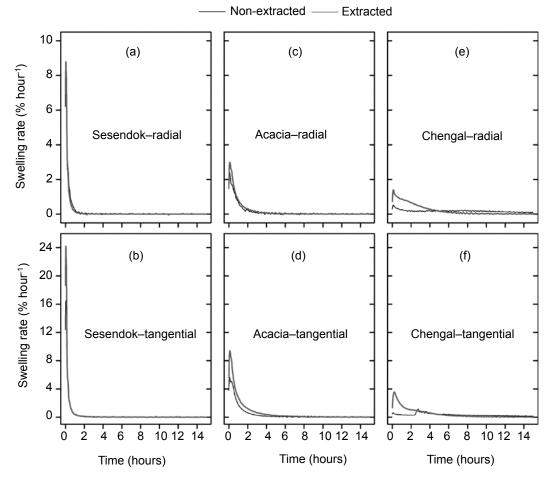


Figure 4 Water swelling rates of non-extracted and extracted (a) sesendok in radial and (b) tangential directions, (c) acacia in radial and (d) tangential directions, and (e) chengal in radial and (f) tangential directions

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extraction in acetone/water solution caused mass loss of 36% of wood for brown rot and 28% for white rot. High amounts of extractives in chengal played an important role in inhibiting the growth of rot fungi. This might be attributed mainly to the hydrophobation and partially to cell wall bulking effects imparted by extractives. The results shown in Table 1 had implied that most extractives might exist in the inner cell wall surface, cell lumen and resin canal, thereby creating a hydrophobic environment to prevent or at least decelerate the entry of water. This can be further affirmed by the slow water swelling (Figure 2) and low moisture content after 12 weeks of fungal incubation (Figure 5a). Some of the extractives might also be located in the cell wall interior, bulking the cell walls and thereby restricting access of fungal enzymes to the cell wall interior (Hill 2006). Rubberwood impregnated with extractives extracted from chengal exhibited a certain degree of resistance to the white rot fungus T. versicolor (Yamamoto & Hong 1988). They found that loss of extractives due to the extraction process using hot water or methanol hardly reduced the ability of chengal wood to resist degradation by white rot, which was apparently different from the great mass loss exhibited in this study. The difference might be due to the harsher extraction condition (water/ acetone at 50 °C for 48 hours) used in this study, causing the complete leaching of extractive from cell walls. As a result, nanopores in the cell walls are enlarged at the water-swollen state so that the enzymes of fungi can access the interior of cell walls easily.

CONCLUSIONS

With increased extractives removal, wood exhibited lower water swelling rate and lower maximum swelling. This might be attributed to the reduction in the access of water to the bulk cell wall. This might also explain the greater mass loss of chengal due to removal of extractives as evidenced by higher moisture content in the extracted wood during

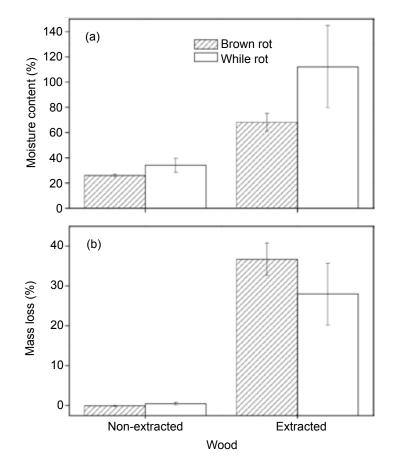


Figure 5 Moisture content (a) and mass loss (b) of non-extracted and extracted chengal wood exposed to brown-rot fungus *Conicohara puteana* and white-rot fungus *Trametes versicolor* for 12 weeks

incubation. The amount of extractives might be a more important factor rather than location of them in the wood microstructures in restraining fungal decay. The relation between durability and cell wall bulking by extractives was weak.

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