CHANGES IN EXTRACTIVE CONTENT ON WOOD SURFACES OF CHENGAL (*NEOBALANOCARPUS HEIMII*) AND EFFECTS ON PERFORMANCE

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AHMAD MN, HALE MD, ABDUL KHALIL HPS & SURYANI S. 2013. **Changes in extractive content on wood surfaces of chengal (*Neobalanocarpus heimii*) and effects on performance**. Chemical composition of decayed surface samples from a dense, durable hardwood from building material partially exposed to weather for 50 years was investigated at various depths within the samples. Results showed that total extractive contents within the surface regions were much higher compared with the inner part. Fourier transform infrared spectra (FTIR) and HPLC studies showed that the composition of extractives varied and was unevenly distributed at different depths. However, holocellulose and lignin contents were 58 and 28% at the surface zone and increased deeper within the sample to 67 and 34% respectively.

Keywords: Extraction process, weathering, deterioration, density profile, holocellulose, lignin

AHMAD MN, HALE MD, ABDUL KHALIL HPS & SURYANI S. 2013. **Perubahan kandungan bahan ekstraktif pada permukaan kayu chengal (*Neobalanocarpus heimii*) dan kesannya terhadap prestasi.** Komposisi kimia pada permukaan reputan sampel kayu keras tahan lama dan tumpat daripada bahan binaan yang sebahagiannya terdedah kepada cuaca untuk tempoh 50 tahun telah dikaji pada beberapa kedalaman sampel. Hasil menunjukkan bahawa kandungan jumlah bahan ekstraktif di kawasan permukaan lebih tinggi berbanding dengan bahagian dalam. Kajian FTIR dan HPLC menunjukkan komposisi bahan ekstraktif adalah pelbagai dan tersebar tidak sama rata pada kedalaman yang berbeza. Akan tetapi, kandungan holoselulosa dan lignin pada permukaan adalah masing-masing 58% dan 28 % dan meningkat kepada 67% dan 34% jauh ke dalam sampel.

INTRODUCTION

Wood is widely used as industrial raw material for construction production. However, during natural weathering of wood, besides discoloration, changes in wood surface texture are most noticeable. The surface becomes rough and fissured and wood constituents will be eroded. The extent of erosion depends on the structure of wood and is fairly uniform in hardwood or more relief-like for softwood (Sell & Feist 1986). Generally, wood weathers rather slowly. If there is no protection by surface finishes and/or building design and deterioration by microorganisms, the depth of erosion of a board exposed to weather is 2 to 5 mm per century for hardwood and 5 to 10 mm per century for earlywood of softwood (Feist et al. 1984, Schulz et al. 1984), although this is somewhat dependent on climatic conditions and wood density. Ultimately, the erosion of wood surfaces during weathering is a result of chemical degradation of wood components by ultraviolet (UV) and visible light followed by mechanical abrasion and dissolution due to rainwater and wind (Müller et al. 2003, Temiz et al. 2005). Under conditions of high humidity and constant wetting, bacterial and fungal degradation may also occur. Therefore, to a large degree, the working life span of exterior wood panels is dictated by the ability of the material to resist degradation from heat, humidity, chemical and UV. The improvement of durability of wood panels and their resistance to these factors can be achieved by application of coating, additive and chemical modification (Chang & Chou 2000, Evans et al. 2002, de la Caba et al. 2007).

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This study described the change in chemical composition and some of the physical properties at the surface of a durable wood species, chengal (*Neobalanocarpus heimii*), after partial exposure to weather for about 50 years.

MATERIALS AND METHODS

Wood sample

Wood specimens were taken from a floor beam of a house of a rubber estate worker in Penang, Malaysia. The beam was about three feet high from the ground and partially exposed to the environment. The house, estimated to be more than 50 years old, was demolished to make way for development. The climate that the wood sample was exposed to was equatorial and characterised by the annual south-west (April till October) and north-east (October till February) monsoons. Temperature was moderated (24–34 °C) by the presence of surrounding oceans. Humidity was high, about 75%, and average annual rainfall was 250 cm (Saw 2007).

Wood sample was identified by light microscopy as *N. heimii* or locally known as chengal. This wood is a naturally durable species (Baharuddin 1986). Microscopic examination of deteriorated/unprotected surfaces of the wood revealed that soft rot decay was the main cause of damage. The extent of surface deterioration was estimated to be about 3 mm in depth on some of the decayed, unprotected faces. In some areas, surface soft rot decay pockets extending deeper into the wood were noted. On a paint protected surface (C in Figure 1), no deterioration was visible.
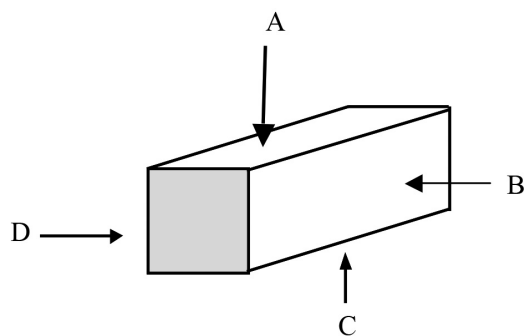

Figure 1   Cross-section of wood block sample (15 cm wide × 8 cm thick × 60 cm long); surface (A) most decayed, (B) and (D) least decayed and (C) protected by paint

Extraction method

Wood sample used was obtained from the sound, inner part of the wood block, approximately 10 mm from decayed surface A (Figure 1). Scots pine (*Pinus sylvestris*) heartwood was also analysed for comparison. Pine samples were obtained from the School of Agricultural and Forest Science, University of Wales, Bangor. Pine samples had been stored at room temperature for about 1 year before use. The wood sample was ground and passed through a 200-mesh sieve and then oven dried. Extractives were removed using standard Soxhlet extraction methods (Anonymous 1988) with slight modification using toluene instead of benzene. Four different solvent mixtures were used (Table 1). The extraction processes were carried out in triplicates. After extraction, solvents were removed from extracted solutions by vacuum evaporation and the extractives were quantified by drying to constant weight. The weight percentage of extractives was calculated and expressed based on the oven dried weight of the wood powder sample.

Cold water extractions were also carried out on the wood powder sample at different periods of time to determine the rate of extractive leaching in water. A 2 g test sample was mixed with 300 mL of distilled water in a beaker. The mixture was allowed to digest at a temperature of 15 ± 2 °C, with frequent stirring until a maximum of 25 days. The material was filtered on a fritted-glass crucible, using suction, washed with cold distilled water and dried to constant weight at 105 ± 2 °C (Anonymous 1993).

Determination of extractive profile at different depths within the deteriorated surface

The extraction process was carried out using method I (Table 1) on wood powder sampled from pieces taken from various depths (0.26, 0.44, 0.7, 0.9, 2, 3.65, 5.7, 7.05, 7.14, 7.23 and 10.00 mm) within the wood block from the decayed side (surface A, Figure 1) and extractive quantities were determined. Fourier transform infrared spectra (FTIR) in the frequency range of 4000–500 cm^{-1} of the ethanol:toluene extractives, each from different depths, were mixed with potassium bromide at a ratio of 1:100 to form potassium bromide discs. Other semi-quantitative differences in extractive contents

Table 1   The four different extraction methods (I–IV) investigated and extraction times

Extraction method			
I	II	III	IV
(1) Ethanol:toluene (1:2 v/v), 4 hours	Toluene:methanol:acetone (4:1:1 v/v), 4 hours	(1) Toluene:methanol:acetone, (4:1:1 v/v), 4 hours	Hot water 90 °C, 3 hours
(2) Ethanol, 4 hours		(2) Hot water 90 °C, 3 hours	
(3) Hot water 90 °C, 4 hours			

were investigated by HPLC using mixture of acetonitrile:water (70:30, v/v) as a mobile phase and a Supelco sil LC-18 column (300 mm length, 3.9 mm diameter). Peaks were detected by UV at 254 nm for extractives from different depths.

Determination of lignin and holocellulose contents

Two methods, namely, a mixture of sulphuric and phosphoric acids, 6:1, v/v (Jayme 1958) and a modified Klason 72% sulphuric acid method (Effland 1977), were used to quantify lignin from wood powder subjected to extraction methods I, II and III. In the Jayme method, 15 mL of a mixture of sulphuric and phosphoric acids were added gradually while stirring into 1 g of extractive-free wood powder sample in a beaker for 1 min. The beaker was kept in a water bath at 35 °C and the mixture was gently stirred for 2 min. The mixture was then added into a beaker filled with 350 mL distilled water. The remaining mixture was washed with 50 mL distilled water into the beaker, which was then boiled on a hot plate for 30 min. Constant volume of solution was maintained using reflux condenser. The mixture was removed from the hot plate and lignin was left to settle for 30 min. The lignin was filtered through a glass filter and oven dried until constant weight. In the Effland method, 4 mL of 72% sulphuric acid was added into a beaker containing 0.24 g extractive-free wood powder. The mixture was placed in a 30 °C water bath, stirred frequently until completely dissolved. After exactly 1 hour, the solution was diluted with 100 mL distilled water. Secondary hydrolysis was carried out in an autoclave at 120 °C for 1.5 hours. The hot solution was filtered off through glass crucible, washed with 250 mL hot water and oven dried until constant weight. Lignin content from similarly extracted pine heartwood samples was also determined for

comparison. The percentage of lignin and holocellulose were determined based on the extractive-free oven-dry weight of wood samples.

Effland method which was more effective and able to completely hydrolysed carbohydrates to produce lignin was later chosen to study lignin contents at different depths within the sample. A similar method used to obtain FTIR spectra for the extractives was also used for lignin.

Holocellulose content was determined following the chlorite method (Wise 1945) but with additions of chlorite at 3, 5 6 and 7 hours. The efficacy of the extraction process was checked by lignin determination of the holocellulose using the Effland method.

Determination of density profile of wood sample

Weathering and soft rot decay will decrease the density of wood surfaces, so quantitative determination of composition at the surface will be biased when making powder samples for analysis. Therefore, wood samples from various sides of surfaces (Figure 2) were cut into specific sizes and then weighed. Dimension (thickness × width × length) and weight of the samples were: (1) 11.87 × 50.36 × 49.01 mm, 22.69 g, (2) 11.16 × 50.09 × 50.07 mm, 18.63 g, (3) 10.71 × 50.19 × 50.26 mm, 22.54 g and (4) 12.98 × 48.43 × 48.99 mm, 25.06 g. Samples were placed in the attenuated total reflectance (ATR) density profile machine (Co[60] scanner) which automatically measured density profile through thickness of the samples. The arrows in Figure 2 indicate the direction of measurement.

Determination of physical properties of samples

To determine surface softening, the depth of penetration was measured using a Pilodyn (6
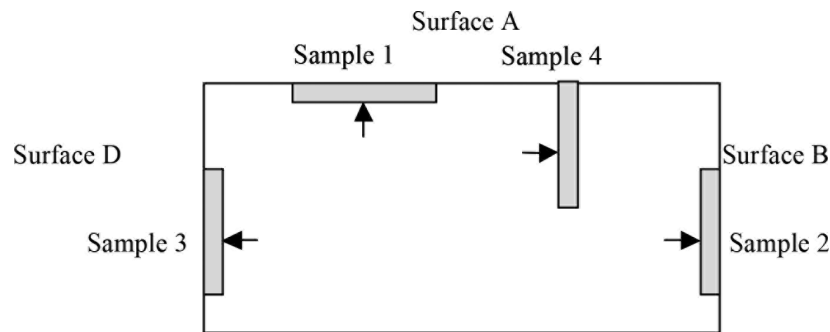
Figure 2   Sample cut positions from surfaces A, B and D for density profile measurement

J spring) on various surfaces of wood, namely, decayed surfaces of various sides, sound wood and inner parts of wood block (about 10 mm below the surfaces).

RESULTS AND DISCUSSION

Extractive content

The extractive contents obtained using different solvent mixtures are shown in Table 2. Method I was most effective for removing extractives from chengal heartwood although the process required a longer time. The extractive content was high (16.1%), similar to that reported by Kishino et al. (1995), i.e. 16.5%. The extractive content of chengal was three to four times higher than Scots pine heartwood (methods I and III).

Removal of extractives from sound wood in hot water alone was remarkably high (14.2%, method IV, Table 2). However, in cold (15 °C) water, removal was a very slow process (initial rate 0.012% hour^{-1}) compared with hot water (equivalent rate 0.197% hour^{-1}) (Figure 3). The rate of loss (based on slope of curve) was not linear and decreased with increasing exposure time. These results indicated that extractives were mobilised with exposure and large amount of extractives that resulted from sufficient water solubility gave long-term resistance.

Extractive content at the wood surface was high and then decreased reaching a minimum at 2-mm depth (Figure 4). Lower corrected total extractive was observed accounting for lower density of wood surface as result of decay. Deeper into the wood the amount increased to a constant value in the sound zone (7–10 mm). This was probably due to the mobility of wood extractives and increase in solubility of wood caused by decay and surface weathering.

The 2-mm depth could be considered the threshold whereby lignin and holocellulose did not deteriorate extensively. In addition, decreases in density, which occurred in the wood surfaces as a result of the decay of structural components of lignin and holocellulose (Figure 5), would lead to relatively higher values for extractive contents. Wood densities at different depths within the sample are shown in Figure 6. Oven-dry density of sound wood, determined from the density profile, was 823 kg m^{-3}. When this density factor was taken into account, extractive contents increased from 19.7 to 26.3%. This showed that results from semi-quantitative analysis of extractives from decayed wood surface were acceptable.

FTIR analyses of the ethanol:toluene extractives revealed a variety of peaks (Figure 7). Many of the peaks were common between individual extracts or present as contaminants. Comparison of spectra showed differences between weathered, decayed surfaces and sound wood deeper into the wood specimens. A small peak at 1173 cm^{-1} was progressively lost closer to the surface and a sharp peak at 1384 cm^{-1} was observed but not for the sound 10-mm sample. The sharp peak at 1611 cm^{-1} declined in intensity and, in the more intensely decayed zones near the surface, a neighbouring peak at 1730 cm^{-1} (carbonyl peak) which increased in intensity closer to the surface was apparent. This might indicate carboxylic acid production.

HPLC studies on the ethanol:toluene extractives (Table 3) from the sound 10-mm depth of the block gave two principal peaks at retention times 3.70 (72.81%) and 3.89 min (27.19%). Extracts from different depths gave a variety of other peaks, few of which had exactly the same retention times. After the peaks were matched up by their proximity and intensity, a

Table 2   Extractive contents (%) obtained from different extraction methods

Extraction method		I		II			III		IV
Wood sample		Chengal	Pine	Chengal	Pine		Chengal	Pine	Chengal
Extractive	(1)	13.5	0.93	12.3	0.62	(1)	12.2	0.68	14.2
	(2)	0.7	0.67			(2)	2.3	3.47	
	(3)	1.9	2.13						
Total		16.1	3.73	12.3	0.62		14.5	4.15	14.2

I: (1) Ethanol:toluene, (2) ethanol, (3) hot water; II: toluene:methanol:acetone; III: (1) toluene:ethanol:acetone, (2) hot water; IV: hot water
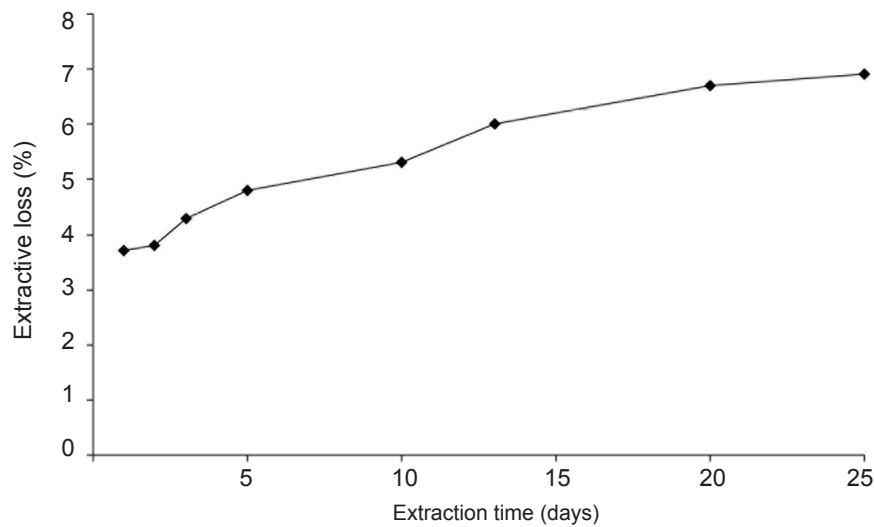

Figure 3   Loss of extractives in a cold water (15 °C) extraction of chengal powder
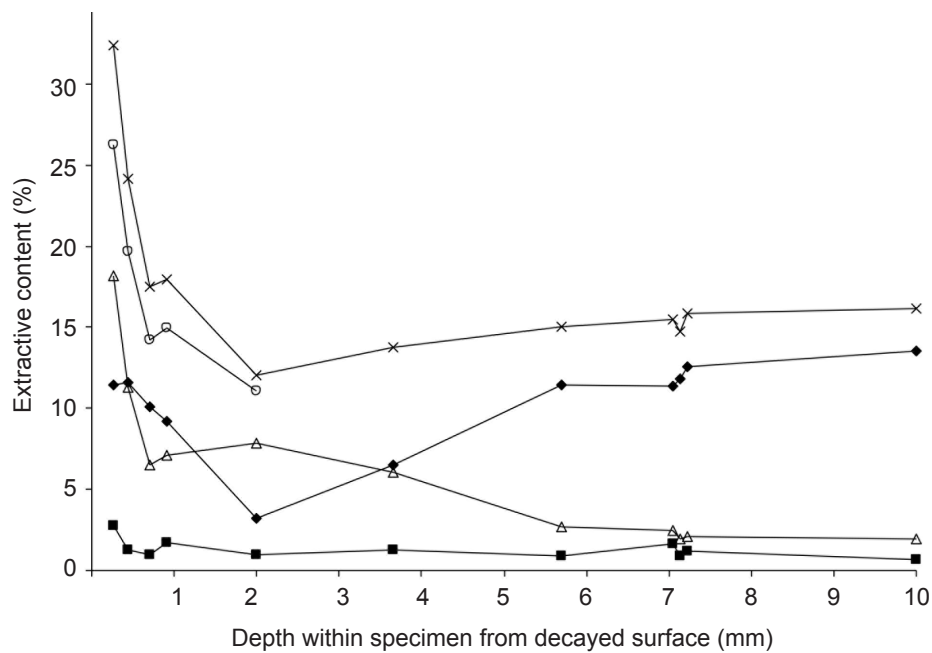

Figure 4   Variation in extractive content from decayed surface A inwards to a depth of 10 mm in the chengal sample; ◆–◆ ethanol:toluene, ■–■ ethanol, △–△ hot water, ×–× total extractives, ○ corrected total extractives accounting for lower density as a result of decay
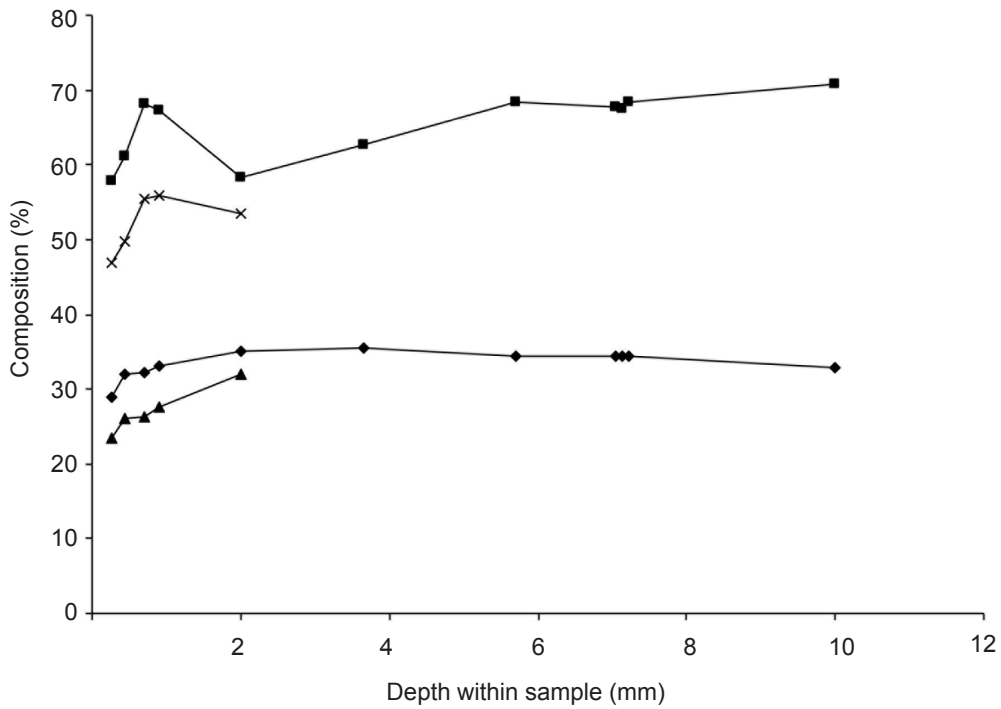
Figure 5   Variation in holocellulose and lignin contents from decayed surface A inwards to a depth of 10 mm in the chengal sample; ■–■ holocellulose, ◆–◆ lignin, ✳–✳ density corrected holocellulose, ▲–▲ density corrected lignin
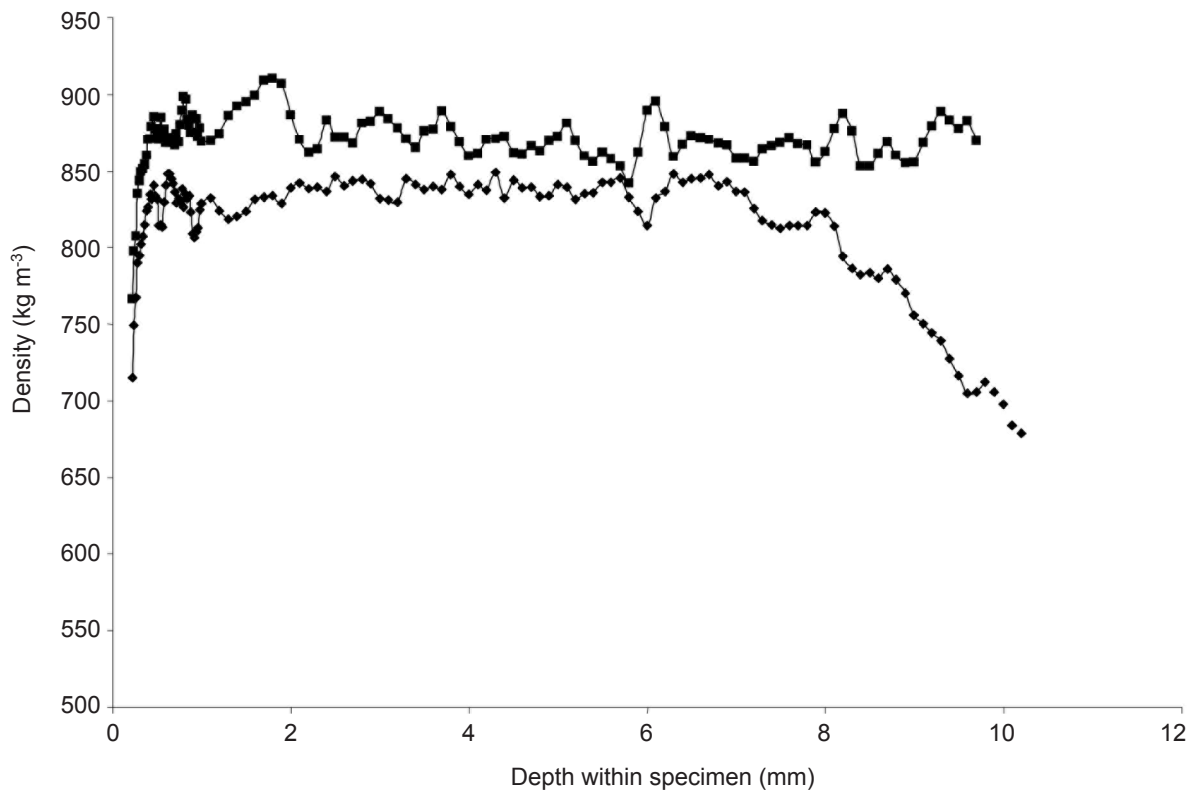

Figure 6   Comparison of density profiles (details in Figure 2) of the decayed edge of sample 1 (◇–◇) and through a sound, undecayed sample, sample 3 (□–□); samples 2 and 4 show essentially the same profiles as sample 3
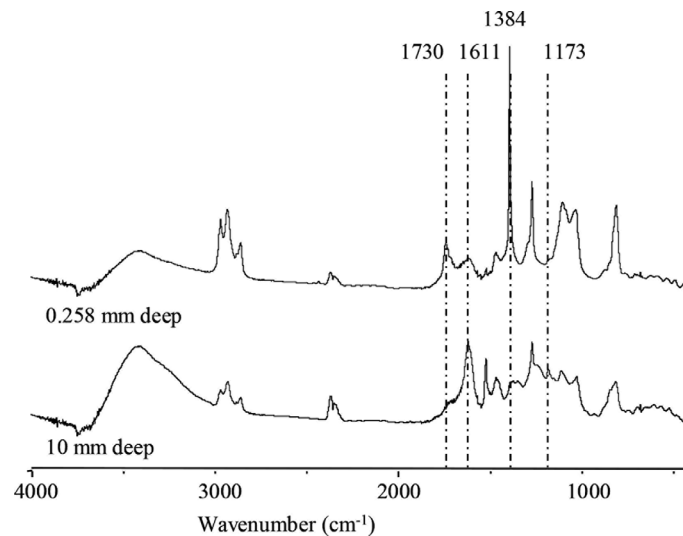
Figure 7   FTIR spectra of traces of ethanol:toluene extractives from wood surface zone (0.258 mm deep) and sound core zone

series of peaks were assigned based on mean retention times in Table 3. The principal peaks (3.70 and 3.89 min) in the 10-mm sample were present in all samples but they both showed considerable variation. From the 10-mm depth to 3.65 mm, the principal peak (3.70 min) declined, while the second peak (3.89 min) remained at similar intensity (20–27%). Closer to the surfaces, considerable variation in retention times occurred. Nearer to the surfaces (2 mm to the surface), relative amounts of different extractive peaks varied considerably and it was difficult to establish any progressive trend. The major peak (3.70 min) declined and then increased at the surface, possibly indicating some depletion from the subsurface and accumulation at the surface. The rest of the peaks appeared to decline at the surface probably as a result of leaching and degradation losses. Combined with total extractive contents, these data did not indicate that the increase in extractive content was a result of the production or accumulation of significant quantities of breakdown products.

Holocellulose and lignin content

Table 4 shows a comparison of lignin contents obtained using the Jayme and the Effland methods. The Effland method seemed to be more effective in completely hydrolysing carbohydrates leaving lignin as an insoluble residue. Lignin contents obtained for pine (26.4%) closely agreed with Pettersen (1984) who reported a

value of 27%. The different methods of extractive removal showed little difference when analysed for lignin content, but there appeared to be some residual extractive included in method II, i.e. toluene:methanol:acetone extraction. Lignin content of chengal was high (34%) in comparison with many wood species (Sjöström 1993). It is a complex aromatic polymer that improves decay resistance to soft and white rots (Deacon 2005).

The effects of successive chlorite additions on the delignification process to determine the holocellulose content are given in Table 5. A maximum of five hourly additions of chlorite:acetic acid proved sufficient to obtain a lignin-free chengal holocellulose (Table 6). Lignin content near the surface was marginally lower compared with deeper into the wood where the amounts were about constant at 34% (Figure 5). Changes at the surface declined at a depth of up to approximately 1 mm. This is in agreement with findings by Feist et al. (1984) and Evans et al. (1996). Lignin decay is normally associated with UV light exposure (Chaochanchaikul et al. 2012) and the rate of lignin decay by soft rot fungi is slow in comparison with holocellulose (Levi & Preston 1965). Lignin is thought to be a major factor controlling soft rot decay (Hale & Eaton 1986). Lignin on the wood surface absorbs UV light which results in preferential lignin degradation. UV-degraded lignin products become solubilised and are washed out by rain (Feist 1988) and this reduction in

Table 3   Semiquantitatives results of HPLC peaks of ethanol:toluene extractives from various depths (mm) of weathered chengal wood

Sample depth (mm)	Retention time (min)					
	2.26	2.46	2.70	3.14	3.70	3.89
	Peak area (%)					
0–0.26	0.60	1.24	0.87	1.56	92.09	3.64
0.26–0.70		5.30	3.69	9.07	47.27	34.67
0.70–0.90		11.52	5.49	27.71	29.77	25.51
0.91–2.00		0.49	0.60	0.63	58.21	40.08
2.01–3.65		12.19	8.52	27.99	30.99	20.11
3.66–5.70	3.20	4.44		29.47	42.98	19.91
5.71–7.05		11.09			63.86	25.05
7.06–7.14		19.33			60.09	20.58
7.15–7.23		19.64			57.94	22.42
7.24–10.00					72.81	27.19

Table 4   Lignin contents (%) of chengal and pine wood

Extraction method	I		II		III	
	Lignin (%)		Lignin (%)		Lignin (%)	
	Chengal	Pine	Chengal	Pine	Chengal	Pine
Jayme method	42.5	34.1	44.7	34.7	43.2	33.6
Effland method	32.4	27.1	33.6	26.1	31.9	26.4
Difference (%)	10.1	7.0	11.1	8.6	11.3	7.2

I: Ethanol:toluene, ethanol, hot water; II: toluene:methanol:acetone; III: toluene:ethanol:acetone, hot water

Table 5   Holocellulose contents (%) of chengal and pine wood

Chlorite added at various time (hours)	Extraction method					
	I		II		III	
	Holocellulose (%)		Holocellulose (%)		Holocellulose (%)	
	Chengal	Pine	Chengal	Pine	Chengal	Pine
3	75.7			67.5		67.3
5	72.5					
6	69.1					
7	67.9	65.2	66.5	66.2	65.4	65.1

I: Ethanol:toluene, ethanol, hot water; II: toluene:methanol:acetone; III: toluene:ethanol:acetone, hot water

Table 6   Residual lignin content of the chengal holocellulose

Chlorite added at various time (hours)	3	5	6	7
Holocellulose (%)	75.7	72.5	69.1	67.9
Residual lignin (%)	2.7	0	0	0

lignin content would encourage soft rot decay. Weathering appeared to effect cellulose less, except in the top surface layer of the wood (Kalnins 1966). The present study showed similar results for the top layer to a depth of 0.438 mm; the content of holocellulose was 58–61% compared with 67% at the layers below (Figure 5).

When total amount of holocellulose and lignin in this region was accounted for, there was a deficit of 13% at the surface which probably constituted a soluble lignin fraction. This caused increases in extractives at the wood surface zones. With the solvent system and column used for HPLC analysis, this compositional difference could not be detected.

FTIR spectra of lignin samples showed that besides difference in intensity deviation, there were no essential spectral differences between lignin obtained from four selected depths within the wood block (Figure 8). This suggested that there were only quantitative effects on lignin due to weathering and effects of the extraction procedure might have masked any changes, or changes might have been more apparent in soluble lignin components.

Density profile

Except for the weathered surface (sample 1) with soft rot decay, the rest of the samples showed no significant differences in density throughout the thickness of the wood samples (Figure 6). A rapid decline at the specimen edge occurred as an artefact of the scanning geometry and was omitted. Figure 9 showed that for the weathered surface, density increased from 650 kg m^{-3} at the edge at a rate of about 60 kg m^{-3} mm^{-1} (0–2 mm) and levelled off at about 850 kg m^{-3} some 3.5–4.0 mm from the surface. The other two surfaces (samples 2 and 3, Figure 2) showed only minor visual surface deterioration and their density values showed no deviation from the average values. The density values were also associated with the greatest changes in extractive contents (Figure 4), their composition (Table 3) and with changes in cellulose and hemicellulose contents (Figure 5), although the greatest changes in these occurred in the outer 1 mm.

Pilodyn penetration

Results from depth of Pilodyn penetration test for the decayed surfaces are shown in Table 7. Inner parts taken from sound zones of the wood block all gave 10-mm penetration depth. Although the surface erosion due to weathering caused density and block hardness to decrease, comparison with the Pilodyn pin depth penetration at the different surfaces showed that most decayed surface gave the least penetration (8 mm) compared with the rest of the surfaces of wood of the same orientation cut from inner sound zones. The use of a less energetic Pilodyn spring might have given more meaningful results.
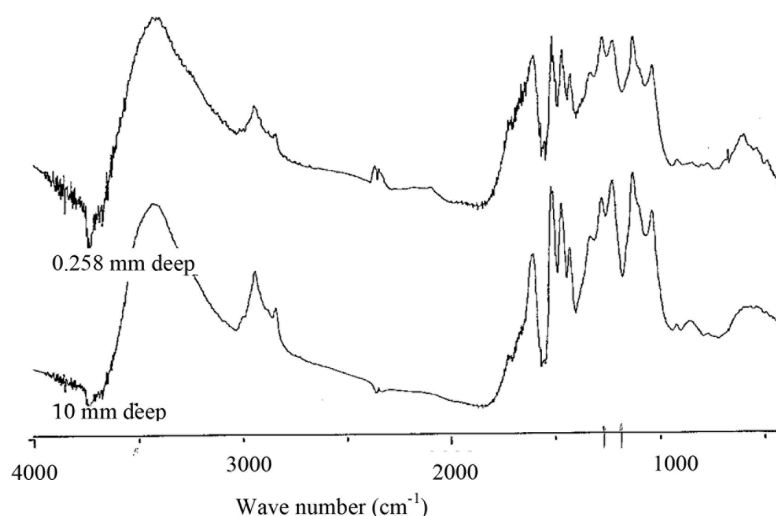

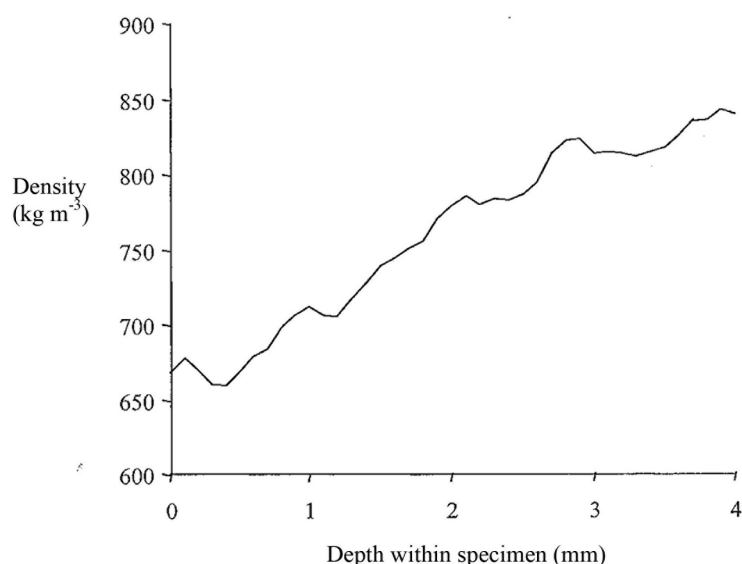**Figure 8**   FTIR spectra of traces of lignin from wood surface zone (0.258 mm deep) and sound core zone

Figure 9   Detailed density profile of decayed surface zone of sample 1

Table 7   Depth of pilodyn penetration into the various wood surfaces

Various part of wood surface	Orientation	Pilodyn penetration depth (mm)
Sound wood, at surface A	Tangential	10
Decayed, at surface A	Tangential	8
Decayed, at surface B	Radial	9
Decayed, at surface D	Radial	10
Inner part of block, side 1	Tangential	10
Inner part of block, side 2	Radial	10
Inner part of block, side 3	Tangential	10
Inner part of block, side 4	Radial	10

I: Ethanol:toluene, ethanol, hot water; II: toluene:methanol:acetone; III: toluene:ethanol:acetone, hot water

CONCLUSIONS

The amount of extractive removed from wood powder is dependent on the type of solvents used. Extraction using the combination of ethanol:toluene (1:2, v/v) was the most effective way for removing extractives, followed by ethanol and finally hot water. Cold and hot water extraction removed water soluble extractives differently at initial rates of 0.012 and 0.197% hour^{-1} respectively. Extractive content was much higher within the surface. This phenomenon was probably due to several factors such as mobility of the extractive towards the surface, decreases in wood density at the wood surface and increases in solubility of extractable materials at the wood surface, probably lignin derived. Decay processes and weathering were probably the reasons for this phenomenon.

Insoluble lignin, and to a lesser extent holocellulose content near the surface, were relatively low compared with deeper into the wood and this was due to weathering by UV rays and rain. This reduction may have predisposed the wood surface to soft rot decay. Density profile measurements on decayed wood are useful to allow calculations of actual wood component losses and indicate a sensitive method for looking at decay profiles in samples.

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