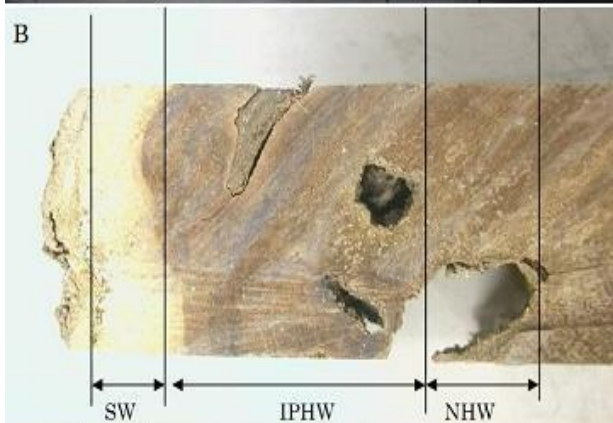
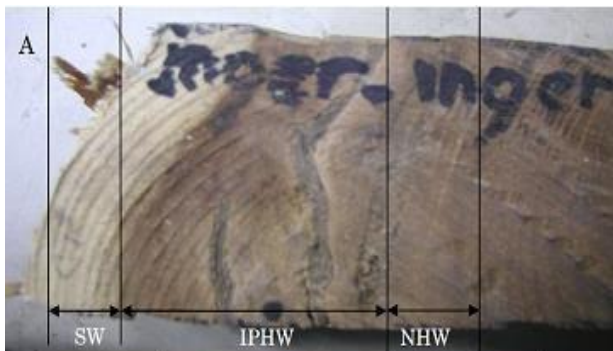


WOOD RESEARCH Journal

Journal of Indonesian Wood Research Society

Volume 9, Number 2, October 2018



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Wood Properties of 5-year-old Fast Grown Teak

Ratih Damayanti, Barbara Ozarska, I Ketut N. Pandit, Fauzi Febrianto, and Gustan Pari

Abstract

Jati Unggul Nusantara (JUN) is one of fast growing plantation teak that has been widely cultivated in Indonesia. This teak has been developed to be harvested after 5 years when its diameter reaches 25-32 cm (diameter at breast high). The diameter of JUN is usually three times larger than the conventional plantation teak (teak cultivated from seed) at the same age, and the same as 30-40 year-old mature teak. Preliminary research was conducted to determine anatomical and selected physical properties of 5-year-old JUN teak, as well as its suitability for furniture production. The results revealed that wood color, texture, and grain pattern of JUN were slightly different from the mature conventional teak. The length of fiber cells was similar as in the mature teak. There were differences in ultramicroscopic structure of JUN: the mean micro fibril angle was narrower, and the crystallites degree was larger. Shrinkage values from green to 12% moisture content were: 0.70 (radial-R) and 1.62 (tangential-T), and from green to oven dry were 1.59 (R) and 3.29 (T). T/R ratio was 2.34. Specific gravity in air dry condition was 0.52. Based on the research results it appears that 5-year-old JUN may be suitable for the production of medium quality furniture products. More research is required to investigate and enhance the properties of JUN for high quality products.

Keywords: Young fast grown teak, anatomical properties, physical properties, super teak, conventional teak wood.

Introduction

Teak (*Tectona grandis* Linn. f) is a popular timber for furniture, construction building, pole boat making and luxurious veneer. Naturally, teak is harvested at approximately 80 years old. Period of rotation in established plantation forests such as in India and Indonesia is 50 to 80 years (Soerianegara and Lemmens 1994). This long rotation in planting causes the price of teak wood to increase significantly due to a limited supply. Iskak (2005) states that the shortage of teak as a raw material is estimated at approximately two million cubic meters per year. Consequently, timber industries that use teak as a raw material face difficulties in its continuous supply (Krisdianto and Sumarni 2006).

This condition motivates the silviculturists to investigate various methods which would allow establishing a shorter rotation and a faster growth teak. One of the methods already developed is through vegetative cultivation, such as tissue culture, bud grafting, and shoot cutting. As a result, the rotation of planting is decreasing from 50-80 years to 20-40 years (Yunianti 2012). These fast grown teaks are becoming a solution to overcome the teak supply scarcity.

In Indonesia, there are many varieties of fast grown teak that have been widely cultivated. Timber communities call this timber "super teak". One of them is Jati Unggul Nusantara (now Jati Utama Nusantara - JUN). The combination of breeding technology and intensive silviculture treatment encourages the teak timber producers to harvest the tree at a very young age, 5-year-old. At this age, the average stem diameter is 20-30 cm which is the

same as 30-40-year-old conventional teak (Ministry of Forestry 2012).

However, there is a common opinion that timbers from fast growing or short rotation have inferior wood properties, mainly in natural durability and timber strength (Irwanto 2006; Kininmonth 1986). A faster growth will produce shorter cells that reduce the wood quality (Brown *et al.* 1994; Panshin *et al.* 1964). No matter how small the changes, it will lead to differences in the macro, micro and ultramicroscopic structures of the wood, which may cause the alterations of material characteristics (Pandit and Kurniawan 2008). This study aimed to determine anatomical and selected physical properties of 5-year-old JUN teak, as well as its suitability for furniture production.

Materials and Methods

Sample Preparation

Four and five year-old JUN were collected in September 2009, located in Balapulang, Central Java Province, Indonesia. While as comparison, 4 and 5-year-old conventional teak were also collected in the same time from Brebes, Central Java Province, Indonesia (at around 10 km from JUN plantation). Wood samples in the disc form measuring 10 cm in thickness were taken from the particular tree heights i. e. bottom, middle and top portions of the corresponding logs for anatomical observation and physical properties investigation. Physical properties as examined were green moisture content, density/specific gravity (SG) and radial as well as tangential shrinkages. Sampling and testing of its physical properties followed the ASTM 2007 D 143-94 Reapproved 2007. Remaining timber was used for furniture manufacturing.

Anatomical Observation

Observation on anatomical structure covered macro, micro and ultra microscopic characters. Macroscopic characteristics involved appearance (including wood discoloration), texture, lustrous, hardness and grain direction. Observation on microscopic structure was prepared by sectioning (Sass 1961) and maceration process (modification of Franklin method in Damayanti *et al.* 2016), covered cells dimension, cells structure (Wheeler *et al.* 1989), and juvenile percentage. The juvenile percentage was determined with the aid of regression curve that related the fiber length to the successive positions of wood segment moving all the way from the pith to the bark in radial direction (Darwis *et al.* 2005) following the growth ring. Furthermore, ultramicroscopic characters assessment covered micro fibril angle, chrySTALLITE degree and cellulose crystallite size using X Ray Diffractometer (Stuart and Evans 1994) from the pith towards bark following the growth ring, taken from the early and late wood portions. Dimension of cellulose crystallite in the cell wall was calculated by Scherrer Formula (Andersson 2006):

$$B_{hkl} = \frac{K \lambda}{\Delta 2\theta \cos \theta'}$$

Distance among cellulose crystallite was estimated by the formula:

$$d = \frac{\lambda}{2 \sin \theta'}$$

where K is a shape factor, the value is 0.9 to determine cellulose crystallite dimension; λ is wave length of Cu; β ($=\Delta 2\theta$) is FWHM value divided by 2 (in radian); and θ' is 2 theta value divided by 2 (in radian).

Quality Assessment as Furniture

Some furnitures from JUN timber were manufactured to evaluate the quality of JUN as raw material for furniture. A set of quantitative criteria as minimum requirements for wood as a raw material for furniture has been established by National Standardization Board (1989). From physical aspect, furniture components need to be produced from timber with minimum strength class III and specific gravity (SG) at least 0.40. Some interviews were also conducted to gain opinion from an experienced technician.

Results and Discussion

Results assesment on wood properties of JUN as furniture material are presented in Table 1. The criteria were determined by Menon and Burgess (1979), PIKA (1979), Pandit *et al.* (2009) and National Standardization Board (1989). Macroscopically, JUN had straight grain direction and sometimes interlocked, the same as 5-year-old conventional teak, while mature teak had majority interlocked grain direction. JUN also had coarse texture, slightly glossy until opaque surface, light color and there was a pattern in wood surface as a result of wood discoloration (secondary heartwood) and multiseriate rays. The latter two characteristics were not observed in conventional teak (Fig. 1). The straight grain direction may ease in furniture processing, and this is a positive aspect because diagonal grain direction usually will reduce wood strength (Pandit *et al.* 2009). The coarse texture in JUN will affect in finishing process such as need more lavish filler or putty, and it may make a problem in sherlak application. Less natural lustrous in JUN requires more effort to increase its luster.

Table 1. Timber properties requirement for furniture and assesment of JUN as furniture material.

Wood Properties	Desired Properties	JUN Properties
Macroscopic structures	Straight grain direction, fine to medium texture, good natural lustrous, good color and appearance	Straight grain direction, coarse texture, slightly glossy until opaque surface, light color and there was a pattern in wood surface due to the presence of wood discoloration and multiseriate rays
Microscopic structures	Medium cell wall thickness, low juvenile wood portion, no crystals, tyloses and silica	Very thin cell wall thickness, 100% juvenile wood portion, no silica, and there was a presence of tyloses
Physical properties	Medium density/specific gravity (SG), high dimensional stability	Shrinkage values from green to 12% moisture content were 0.70 (radial-R) and 1.62 (tangential-T), and from green to oven dry were 1.59 (R) and 3.29 (T). T/R ratio was 2.34. Specific gravity in air dry condition was 0.52. In conclusion, JUN has medium density/SG and rather good dimensional stability



Figure 1. Wood discoloration as a result of secondary heartwood in 5-year-old JUN; compared to 5-year-old conventional teak where the discoloration or secondary heartwood formation was not observed (arrow).

The parts of furniture intended to accept loads, either continuously or intermittently. These loads are evenly distributed including on the connection. Thus, although the strength is important, it is not considered necessary really very strong raw material. Furthermore, wood strength usually associated with wood density, in consequence, very strong wood means very heavy timber. Furniture made of heavy wood is generally less desirable because it is difficult to move. Besides a complicate removing, heavy timber also causes rapid blunting of the knife. Wood with dry oven density in approximately 0.5 g cm^{-3} has proven to be quite good for furniture (Menon and Burgess 1979).

Air dry density of JUN was 0.52 g cm^{-3} with specific gravity 0.48, and it makes 5-year-old JUN will quite ideal to be used for furniture. Although it is advisable to use a timber with larger specific gravity for products that endure heavy load, 5-year-old JUN can be used to manufacture furniture for home and office, such as desks, cabinets, shelves, including bookcases. So far, the manufactured product is strong enough to support loads.

Wood for furniture should be easy to sawn, planed, polished or drilled (Menon and Burgess 1979). In this study, machining properties were not studied quantitatively. Wood surfaces should be smooth with no tear grain that will result in a hairy surface. Qualitatively, at planing process, JUN was easily planed and produced an even surface. It was possibly due to the straight direction of wood grain and the smaller crystal size. Selulose crystallite dimensions for JUN were 5.9 nm (thickness) and 17.78 nm (length) with distance among crystallite was 0.3913 nm, while, thickness, length and distance of crystallite selulose for conventional teak were 6.36 nm, 23.88 nm, and 0.3938 nm, respectively. In

sawmills process, rough surface of JUN made it rather difficult to sawn. The technician called the wood processing properties of JUN was like *Dryobalanops* wood, and it was possible because there was a similarity in their vessels pattern.

Juvenile portion both in JUN and conventional teak were 100%. This high juvenile wood percentage will decrease its quality as furniture. Characteristics of juvenile wood generally have a low density, high moisture content, and high longitudinal shrinkage; making it is easy to deform. The most feared nature of juvenile wood is disabilities called brittle, especially for structural timber, so its use as component for construction is not recommended (Anisah and Siswamartana 2005). For the furniture industry, wood with a high percentage of juvenile wood would also tend causing a lot of problems during the processing. However, in the same age, JUN has more declivous regression line (Fig. 2). It meant that JUN may reach a wood maturity faster than conventional teak wood, however it needs more study on the older ages. Furthermore, the narrower MFA of JUN enables JUN to overcome the high longitudinal shrinkage in common wood. MFA of JUN was 22.09° while conventional teak was 25.29° . Another study stated that MFA of 7-year-old fast growing teak from Penajam, East Borneo, was 23.29° , decreasing from pith to bark, while MFA of 7-year-old conventional teak was smaller, 22.05° , with the same pattern (Krisdianto 2008). The narrower MFA in JUN was predicted as a result of shoot cutting cultivation technique from mature parent trees and a combination with compound support root (Fig. 3) that enables JUN has narrower MFA without having broken in the early growth.

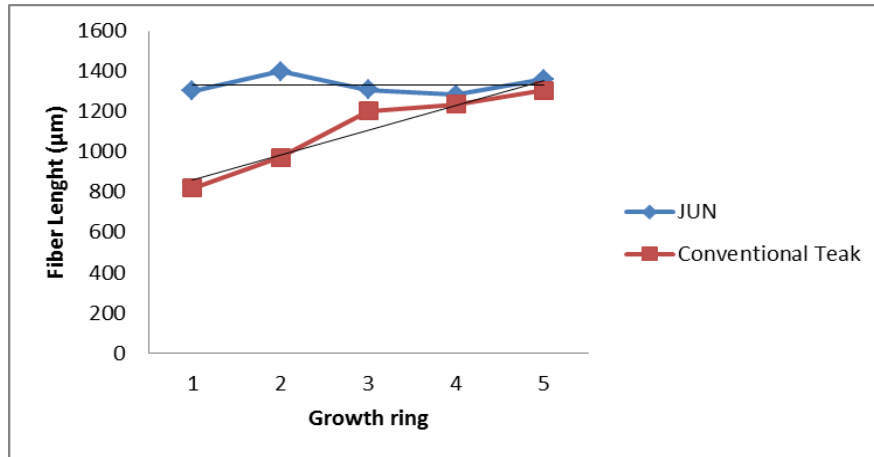


Figure 2. Regression curves of the fiber length from pith towards bark for 5-year-old JUN and conventional teak. It appears the trend is still pointing upwards, yet there is a constant point. It can be seen that the fiber length of JUN started above 1200 µm (similar as in mature teak), and fiber length addition of JUN is more sloping than conventional teak. JUN may reach a wood maturity faster than conventional teak wood.



Figure 3. Compound support root in JUN enables JUN has narrower MFA without having broken in the early growth.

Table 2. Radial (R) and tangential (T) shrinkages of 4- and 5-year-old JUN and conventional teak wood

Type of Teak	From green to 12% moisture content		From green to oven dry		T/R ratio
	R	T	R	T	
JUN	0.70	1.62	1.59	3.29	2.34
Conventional teak	1.88	3.03	2.77	4.43	1.68

Qualitative testing for JUN hardness showed that JUN had low hardness. Positively it may ease the wood processing including the drilling, however negatively its strength on nails holding may rather weak. Weakness in nails holding and easier drilling properties were due to the very thin wall of JUN's cells.

Timber for furniture raw material should not contain too much wood extractive such as resin, and also silica because it will accelerate blade blunting. Since JUN was harvested in very young age, macroscopic observation showed that extractive and silica content was low, and it will ease in processing.

Timber with high shrinkages will not be preferred for any utilisations. Dimensional alterations will cause distortion in furniture component, hard to pull the drawers, hard to open cupboard's door, and sometimes open wood joints (Menon and Burgess 1979). Average shrinkages values from green to 12% moisture content and from green to oven dry for 4 and 5-year-old JUN and conventional teak wood presented in Table 2. T/R ratio for JUN was 2.34 while conventional teak was 1.68. The conventional teak wood had very good dimensional stability, whereas T/R ratio of JUN that was above 2 but under 2.5 showed that JUN had medium stability. An effort, such as an appropriate drying treatment or quality enhancement treatment such as densification and impregnation (Corryanti and Muharyani 2018) absolutely is needed to increase the wood dimensional stability of 5-year-old JUN.

Particular attention, however, must be applied when the wood furniture will be used in air-conditioned room. Therefore, wood with low shrinkage is ideal for furniture production. Changes in water content of the dried wood can be minimized by using proper coating varnish, paint, or even a plastic sheet. The latter method is the latest development in wood protection techniques. If possible, the board should use radial board because it has a smaller shrinkage (Menon and Burgess 1979), and the radial shrinkage of JUN was very high due to the wider rays, higher crystallite degree, and narrower micro fibril angle (MFA). Crystallite degree and MFA of JUN was 43.89% and 22.09°, respectively, while conventional teak was 40.32% and 25.29°, respectively.

In conclusion, regarding to its properties, 5-year-old JUN may be used as raw material for furniture. Requirements in terms of strength, wood processing, wood density, and dimensional stability were met the minimum limit even though there were weaknesses in nails holding strength and lavis finishing. Teak popularity enables JUN with a lower hardness and lighter wood properties sometimes preferably to use because easier to processed and moved. In terms of appearance, its quality as a luxury product will decrease, especially for furniture that requires wood beauty, as of, JUN more suitable for making light-colored furniture that is preferred by certain consumers. However, there is a possibility that its appearance will appeal if used at an older age.

Conclusions

Efforts to accelerate teak growth in Jati Unggul Nusantara led to changes in the anatomical structure of wood and some properties. Until the age of 5 years, input technologies given can improve the properties of the wood to the ultrastructural level where the mean micro fibril angle was narrower, and the crystallites degree was larger. The properties of JUN will facilitate JUN as raw material for furniture, although there was a decrease in appearance and wood texture.

Acknowledgements

Authors would like to thanks to PT. Setyamita Bakti Persada and Unit Bagi Hasil KPWN Ministry of Forestry, Indonesia, especially to Bapak. Hariyono Setiyono, Bapak. Rafik and Bapak. Baghya Siregar for their help in wood sample collection.

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Measurements of Inorganic Materials and Acidity in Plantation Teakwood

Ganis Lukmandaru and Rudy Nur Hidayah

Abstract

Information concerning ash materials and acidity (pH values) within the wood of teak (*Tectona grandis* L.f.) plantations is especially limited. Samples taken from the stands of Perhutani plantation (Madiun and Randublatung) and community forests in Kulon Progo (Temon and Kalibawang) were analyzed to determine the ash content, metallic elements constituting the ash fraction, as well as pH values. The ranges of ash content were 0.55-3.88% whereas acid insoluble ash content (silica/silicates) were 0.12-2.45%. The main four inorganic elements in wood were assayed by atomic absorption. The levels of these metals ranged from 340-4774 ppm for calcium, 17-4399 ppm for potassium, 143-1676 ppm for magnesium, and 0-247 ppm for iron. Further, the pH values varied from 5.33-7.25. Differences of inorganic variables and pH values in wood were found between trees of different growth-site and radial position. The variation among the different sites was significant in the contents of silica, calcium, magnesium, potassium, and sodium while the effects of radial direction were significant in the silica and potassium levels. Ash content was positively correlated with acid insoluble ash ($r = 0.77$) and potassium content ($r = 0.47$). Furthermore, pH values were positively correlated with the magnesium content ($r = 0.65$) and negatively correlated with potassium ($r = -0.49$) and sodium contents ($r = -0.55$). A description of the chemical properties of the soil, however, was not sufficient in determining whether there was a relationship between the levels of metal elements or pH values in the wood and in the soil.

Keywords: *Tectona grandis* L.f., ash content, silica, inorganic element, pH value.

Introduction

Teak (*Tectona grandis* L.f.) is one of the most important timbers in Indonesia because of its favourable physical and mechanical properties, combined with its high natural durability, high weather resistance, and beautiful grain. Thus, the wood is suitable for multipurpose use, from outdoor constructions to small handicrafts. On the basis of trading volume, teak wood is the number one for hardwoods in the world. As a consequence of high demand, besides teak plantations managed by the state-owned company Perum Perhutani, an increase in the quantity of teak produced in community forests or farmland has occurred in Indonesia within the last two decades.

Recently, studies on teak timber mostly focus on the basic properties and natural durability of wood from fast-growing or younger trees e.g. Basri and Wahyudi (2013), Hidayati *et al.* (2014), Lukmandaru (2013), Marsoem (2013), Marsoem *et al.* (2014). With regard to wood chemistry, other important information which also affects the wood utilization include the existence of inorganic materials and the acidity (pH) of the wood. These properties have been reported to affect the cutting (Shmulsky and Jones 2011), gluing and coating (Adamopoulos *et al.* 2005; Pedieu *et al.* 2008), and discoloration of the wood (Minato and Morita, 2005; Mayer and Koch 2007).

Several studies have been conducted to investigate the inorganic materials and acidity of teak wood from various origins (Kjaer *et al.* 1998; Lukmandaru *et al.* 2009; Ola-Adams 1992; Windeisen *et al.* 2003). Unfortunately, the data from teak plantations in Indonesia is still limited. Thus,

the primary objective of this study was to determine the amounts of total ash, individual elements, and the acidity of teak wood from the stands of Perhutani plantation and community forests for scientific and technical interests in wood processing. The other purposes of this study were analyze the relationship between the content of the inorganic variables and acidity of the wood, as well as the chemical properties of the soil.

Materials and Methods

Sample Materials

Wood samples were obtained randomly from the farmland stands and the Perhutani plantation. Four field collection sites (Fig. 1) were selected; these sites were known to yield a large annual harvest and had different ecological attribute. Tree samples from Perhutani plantation stands were felled from Madiun Forest Management Unit (East Java Province) and Randublatung Forest Management Unit (Central Java Province) at a class age of VI. Tree samples from the community forest were cut from Kulon Progo Regency i.e. Kalibawang and Temon village at a diameter class of 20-35 cm. This tree selection was based on the cutting period generally practiced in the related sites.

At each site, three trees with similar features were selected, and a 5-cm-thick disc was collected at approximately breast height from each tree. The test specimens were taken successively from sapwood to heartwood, and divided into three sections diametrically i.e. sapwood (SW, ca. 0.5 cm from the bark), outer heartwood (OH ca. 0.5 cm from the heartwood-sapwood boundary),

and inner heartwood (IH, ca. 2 cm from the pith) (Fig.2). Each part from two opposing radii was converted into wood meal by drilling. The meals from two opposite radii were then combined to form a single sample in order to minimize any variation between radii. The condition of the sites and tree characteristics are described in Table 1.

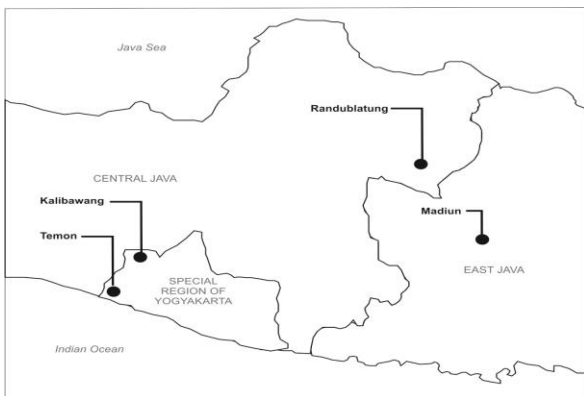


Figure 1. Geographic distribution of sampling sites.

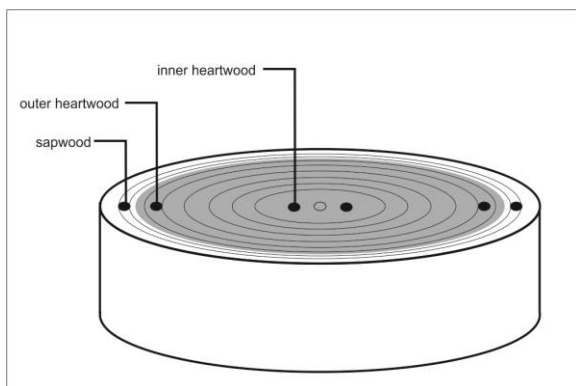


Figure 2. Schematic diagram of division of wood discs for the analysis of inorganic materials and pH value.

Inorganic Materials Analysis

Measurements of ash content and acid insoluble ash content (AIAC) were conducted according to the ASTM D-1102-2002 and TAPPI T244 om-88 standard method, respectively. The filtrates from the AIAC measurement were prepared for elements analysis. Measurements of potassium (K), calcium (Ca), magnesium (Mg), sodium (Na), and iron (Fe) were carried out using an Analytik Jena 300 series atomic absorption spectrophotometer. Duplo measurements were made for each part. The results were shown according to the initial mass of dry wood.

Measurement of pH Value

The acidity of the wood was determined by its pH value. Wood powder (1 g per part eq. dry weight) was suspended for 48 hours in distilled water (20 mL). The pH of the filtrate was measured with a pH meter (OAKTON pH tester). Three measurements were made for each part.

Data Analysis

The testing for normality of data was conducted first then the data were analyzed (General Linear Models Procedure) by two-way analysis of variance (ANOVA) followed by Duncan's multiple range test ($p = 0.05$). The relationships between the independent variables were studied with a Pearson's correlation analysis. All statistics were performed with Excel (MS Word 2007) and SPSS-Win 16.0.

Table 1. Characteristics of the growth-sites and trees felled for sampling.

Origin	Kalibawang	Temon	Madiun	Randublatung
Altitude (m asl)	770	15	120	140
Annual rainfall (mm/year) ^a	2.227.8	1.966	1.600 - 2100	1300-2000
Parent rock	Volcanic	Limestone	Limestone	Limestone
Soil type ^b	Latosol, rocky	Mediteran, clay	Mediteran, clay	Grumusol, clay
- total potassium (%)	0.15	0.06	0.02	0.24
- total calcium (%)	0.58	6.14	0.67	6.09
- total magnesium (%)	0.45	0.51	0.35	0.50
- pH value	6.50	7.82	5.82	7.60
Dbh (cm)	20.7 – 23.4	20.8 – 34.2	36.3 – 50.3	54.8 – 67.7
Annual ring number	10 – 12	13 – 17	61 – 65	67 - 70
Heartwood percentage	61 - 64	60 - 65	77 - 83	94 - 98

Note :

^a data were taken based on the annual report by local Badan Pusat Statistik (Statistics Indonesia)

^b the determination of total calcium, potassium, and magnesium was conducted by extraction with HNO₃ and HClO₄ preparation method.

Results and Discussion

The quantification of inorganic contents and pH values based on growth-site factor is summarized in Table 2. It is noted that the levels of individual metals varied considerably, as can be seen from the high standard deviations. ANOVA was used to separate variation sources resulting from differences within growth sites and radial position which is presented in Table 3. The ANOVA indicated that there was significant interaction between the site and radial position in regards to the ash content and pH value. The variation among the different sites was significant in the contents of AIAC, Ca, Mg, K, and Na while the effects of radial direction were significant in the AIAC and K levels. As the data was not a normal distribution, Fe content was not further analyzed by ANOVA.

Ash and Acid Insoluble Ash Contents

In this study, dry ashing was conducted for removal of organic fraction. AIAC representing silica or silicates in the ash fractions was obtained by dissolving the remained ash with hydrochloric acid. The range of ash content in previous works were 0.97-4.10% for teak heartwood grown in Java (Lukmandaru 2010; 2011; 2012) or 0.7-2.8% for teak wood from Brazil (Polato *et al.* 2005). Furthermore, the range of silica contents in teak from various origins recorded in previous works were 0.18-1.40% (Kjaer *et al.* 1998) and 0.4 % from Indonesia (Martawijaya *et al.* 1981). For sapwood and heartwood region, the ash content ranged from 0.73 to 2.46% and 0.71 to 3.83%, respectively whereas the AIAC ranged from 0.12 to 1.34% and 0.20 to 2.45%, respectively. Thus, the corresponding values of ash content

in this study are in the previously reported range whereas AIAC levels are slightly higher than those previously reported and probably reflect normal geographic variation. On the basis of total weight of ash content, 16.4-54.4% in the sapwood and 28.1-63.9% in the heartwood are attributed to the silica content. Although silica is impervious to insects and marine borers (Cookson *et al.* 2007), the timbers that contain more than about 0.3% of silica cause unduly rapid blunting of saws (Shmulsky and Jones 2011). Thus, in regards to wood utilization, these properties are disadvantage and tree selection should be applied to find trees with low silica content in the future.

Inorganic Elements

Ash fractions from teakwood have not been studied extensively, especially for their intra- or inter-tree variation. In general, the major elements in the wood is calcium (about 80%) and potassium and magnesium are the other predominating elements (Fengel and Wegener 1989). Atomic absorption analysis of the ash residues revealed the presence of 5 individual metals. On the basis of the sites, varied tendencies of element content were observed. The ranking of observed metals from high to low content was as follows : K>Ca>Mg (Kalibawang), Ca>K>Mg (Temon and Madiun), and Mg>Ca>K (Randublatung). Previous study in a partially black-streaked heartwood of teak showed a Ca>K>Mg trend in the normal heartwood and K>Ca>Mg trend in the sapwood (Lukmandaru *et al.* 2009). In other species, such as *Robinia pseudoacacia* (Adamopoulos *et al.* 2005) and some conifers and hardwoods from Japan (Tsuchiya *et al.* 2009), the highest exhibited content was Ca, followed by K and Mg.

Table 2. Contents of inorganic materials (% of oven-dry wood) and pH value of teakwood trees from different sites.

Parameter	Kalibawang			Temon			Madiun			Randublatung		
	Min.	Max.	Average (sd)	Min.	Max.	Average (sd)	Min.	Max.	Average (sd)	Min.	Max.	Average (sd)
Ash content (%)	0.55	3.88	2.61 (0.98)	0.62	1.64	0.83 (0.32)	0.71	2.82	1.39 (0.69)	0.88	2.36	1.59 (0.53)
Ash insoluble acid content (%)	0.41	2.45	1.22 (0.77)	0.12	0.46	0.38 (0.28)	0.22	1.52	0.67 (0.50)	0.20	1.76	0.81 (0.44)
Calcium (ppm)	356	1823	857 (600)	340	2166	861 (615)	1808	4774	2909 (1231)	607	1411	907 (281)
Potassium (ppm)	711	4399	1670 (1240)	197	1669	786 (652)	185	2211	813 (842)	17	1035	434 (450)
Magnesium (ppm)	143	1676	744 (411)	411	987	629 (171)	363	844	624 (141)	1094	1559	1323 (165)
Iron (ppm)	0	93	22.66 (36.62)	0	1	0.11 (0.33)	0	247	29.22 (81.81)	0	151	16.77 (50.33)
Sodium (ppm)	250	1390	757 (345)	273	1305	554 (335)	27	275	113 (88)	4.32	20	13 (4)
pH value	5.33	6.49	5.80 (0.35)	5.64	6.11	5.86 (0.18)	5.84	6.78	6.20 (0.41)	6.62	7.25	6.95 (0.34)

Note : min. = minimum value, max. = maximum value, sd = standard of deviation

Inorganic material amounts are expressed in percentage and part per million (ppm) based on dry weight of wood prior to ashing

Table 3. Different-site and radial direction analysis of variance in inorganic materials and pH value

Source of variation	df	Mean square						
		Ash content	Acid insoluble ash content	Calcium	Magnesium	Potassium	Sodium	pH value
Site (A)	2	4.9**	1.1**	9311611.7**	999054.8**	2111141.9**	1131189.3**	2.5**
Radial direction (B)	2	2.3**	1.0*	34658.3	24411.3	6036139.8**	62261.8	0.3*
A x B	4	0.7*	0.4	580784.7	43917.4	127197.2	62401.6	0.2**
Error	18	0.2	0.2	630172.0	69026.5	388625.1	59029.3	0.1

Note : df = degree of freedom * significance at the 5 % level ** significance at the 1 % level

The black-streaked heartwood grown in Java was reported to have contents of Ca, K, Mg, and Fe of 760-2500 ppm, 810-1950 ppm, 290-460 ppm, dan 30-55 ppm, respectively (Lukmandaru *et al.* 2009). Teakwood from Nigeria had a metal concentration ranging between 1130-2020 ppm for Ca, 3290-5980 ppm for K, and 480-1240 ppm for Mg (Ola-Adams, 1992). Ca content for teakwood from various provenances (India, Indonesia, Ghana, dan Mexico) was found to range between 2200-5800 ppm (Kjaer *et al.* 1998). Thus, these results in this experiment are still in the range of those above mentioned. However, Fe was distributed only in several samples. For instance, out of 3 individuals of Temon or Randublatung, Fe was detected in only one individual. The exact reason for this is unknown but is probably related to the limited experimental technique used for detecting. Na content was not compared as no published work for Na content teak is available to the best of our knowledge.

Effect of Site and Radial Direction on Inorganic Contents

Inorganic materials enter the tree through the root system and are transported to all tissues within the growing tree. Variations may normally be affected by growth site. Duncan test (Fig. 3) revealed that the highest ash content was measured in the heartwood samples of Kalibawang (3.62%) and the lowest levels were in the samples of Temon (outer dan inner heartwood) and Madiun (inner heartwood).

It is assumed that the levels of inorganic materials is higher in the sapwood for movement of minerals from the wood to younger plant tissue. In this experiment, that trend was observed only in the samples of Temon and Randublatung.

The wood of Kalibawang was by far the richest in AIAC (1.33%) compared to the other samples (Fig. 4). For inorganic elements, the wood of the Perhutani plantation (Madiun and Randublatung) contained more Na than the wood of the community forest in Kulon Progo by Duncan test (Fig. 5). The highest Ca amount (2909 ppm) was observed in the Madiun samples whereas the highest Mg amount (1323 ppm) in the Randublatung samples. Ca and Mg belong to the alkali earth metals (IIA) whereas Na and K belongs to the alkali metals (IA) groups in the periodic tables. The differences of age of tree and site factors in this study might account for this phenomenon as the trees would respond differently related to those metal groups.

The inorganic composition of the wood may reflect both the physiological changes (differentiation, aging, heartwood formation) and environmental conditions. By evaluating the soil properties (Table 1), the association with the wood properties seemed to be unclear. For instance, Kalibawang soil did not show the highest total Mg content but the wood samples did. The same case was also observed in the Madiun samples. Further studies should be conducted using larger samples in order to reduce the high variation in the same stand as well as to confirm whether the phenomenon above was systematic or coincidental.

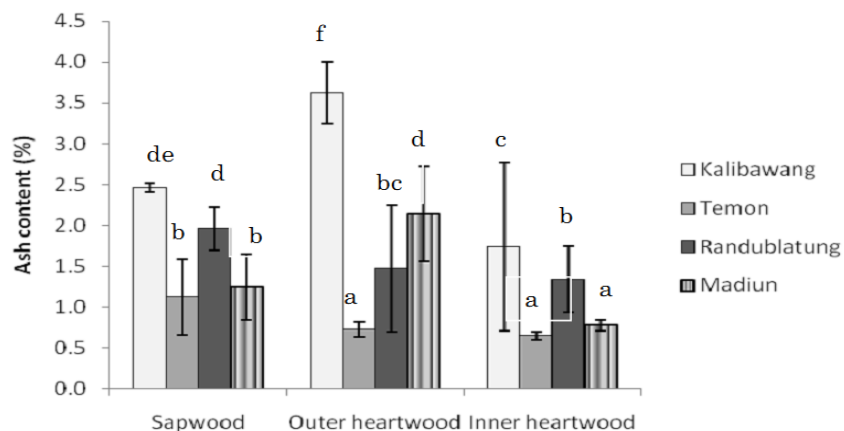


Figure 3. Ash content of teakwood by different sites and radial position. Average of 3 trees, with the standard deviation in the error bar. The same letters are not significantly different at $p < 5\%$ by Duncan's test.

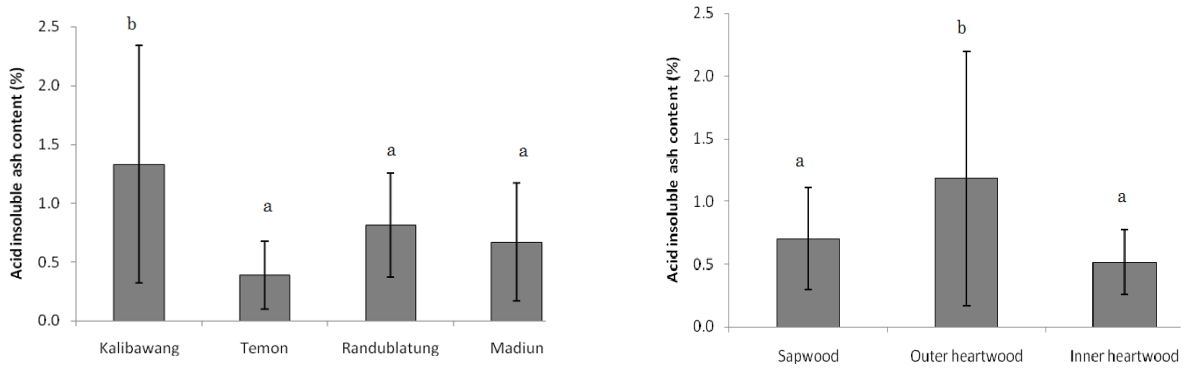


Figure 4. Acid insoluble ash content content of teakwood by different sites and radial position. Average of 3 trees, with the standard deviation in the error bar. The same letters are not significantly different at $p < 5\%$ by Duncan's test.

The woods in Kalibawang were the richest in ash content, AIAC, and K (Fig. 6). Previously, these variations in teak trees were attested to by Ca analysis performed in various provenances (Indonesia, Ghana, dan Mexico), where the values were affected by site factor (Kjaer *et al.* 1999). To estimate the availability of metals, top soil examination at each sampling site was also conducted (Table 1). However, a simple description of soil chemistry as

a feature for interpretation was not sufficient. There is probably another factor that affects the metal ion content of wood beside the chemical composition of underlying bedrock such as climate or other environmental conditions (Fengel and Wegener 1989). Within growth-site factor, besides parent rock and soil type differences, the comparatively high average annual rainfall and the altitude of Kalibawang could be the cause of that phenomenon.

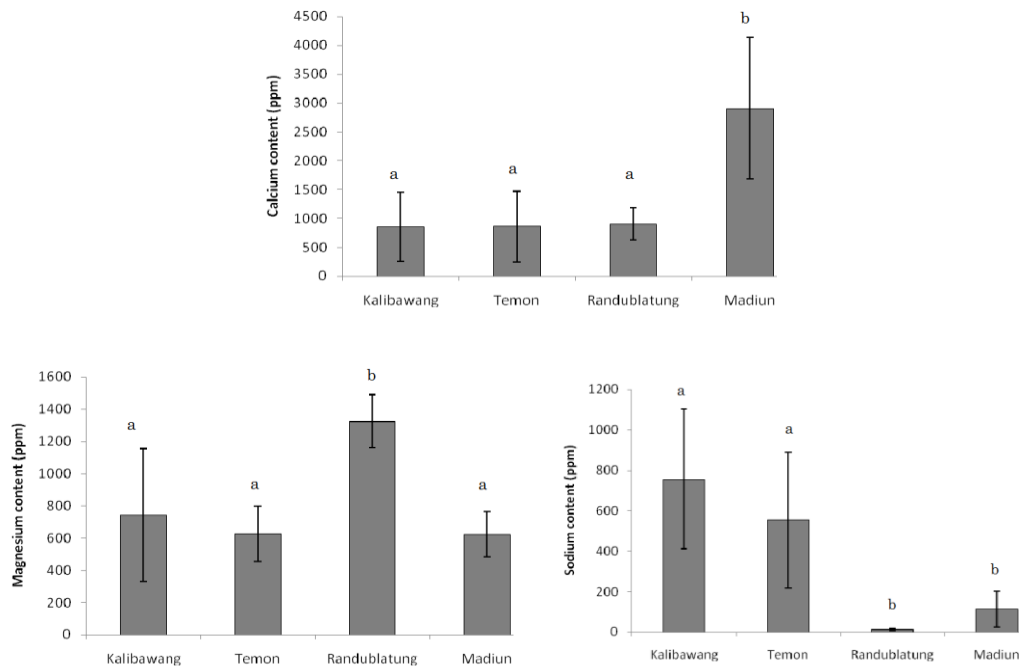


Figure 5. Calcium, magnesium, and sodium content of teakwood by different sites. Average of 3 trees, with the standard deviation in the error bar. The same letters are not significantly different at $p < 5\%$ by Duncan's test.

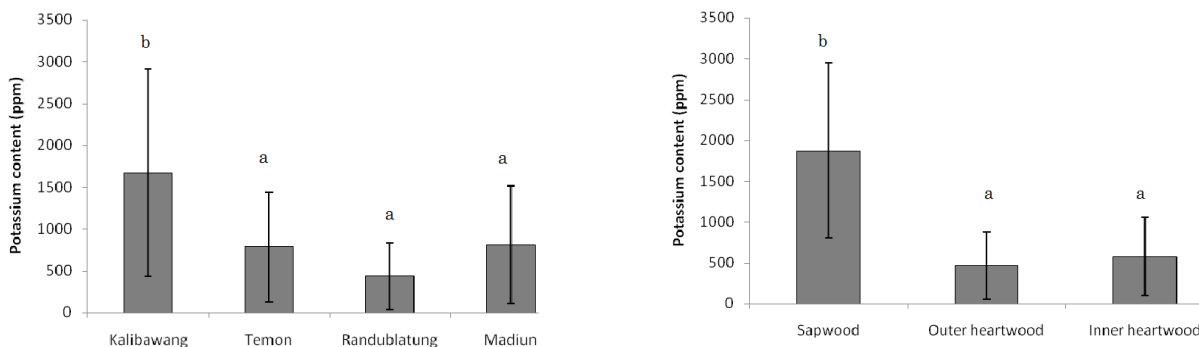


Figure 6. Potassium content of teakwood by different sites and radial position. Average of 3 trees, with the standard deviation in the error bar. The same letters are not significantly different at $p < 5\%$ by Duncan's test.

With regard to radial distribution of inorganic materials, the levels were highest in the outer heartwood for AIAC and in the sapwood for K (Fig. 6). On the basis of radial pattern of trace elements, the former is in Type 3, those whose highest levels are attained at the borderline between sapwood and heartwood while the latter is in Type 1, those whose concentrations fall suddenly from the sapwood to the heartwood (Okada *et al.* 1993). Further, it has been mentioned that alkali metals mostly fell into Type 1 or 3. The abrupt changes in AIAC amounts around the

sapwood-heartwood boundary are possibly related to the heartwood formation process. Of the five metals which were measured, only K was significantly different with respect to radial position. The high concentration of K presumably is related to the cambial zone or differentiating zone in the xylem which may transport large metal contents. The lack of significant differences for all elements in the outer (mature zone) and inner heartwood (juvenile zone) is probably due to the absence of living tissue, making physiological reactions limited there.

Tabel 4. Pearson's correlation coefficients (r) between inorganic substance parameters and pH value in the sapwood and heartwood of teak.

Properties	AIAC	Ca content	Mg content	K content	Na content	pH value
Ash content	0.77**	-0.12	0.10	0.28	0.27	-0.17
AIAC		-0.17	0.15	0.15	0.15	-0.20
Ca content			-0.19	-0.01	-0.35*	-0.05
Mg content				0.12	-0.46**	0.65**
K content					0.34*	-0.08
Na content						-0.52**

Tabel 5. Pearson's correlation coefficients (r) between inorganic substance parameters and pH value in the sapwood of teak.

Properties	AIAC	Ca content	Mg content	K content	Na content	pH value
Ash content	0.71**	-0.38	0.43	0.33	0.03	0.08
AIAC		-0.40	0.57	0.48	-0.16	0.01
Ca content			-0.14	0.08	-0.31	0.11
Mg content				0.17	-0.56	0.44
K content					0.09	-0.36
Na content						-0.67*

Tabel 6. Pearson's correlation coefficients (r) between inorganic substance parameters and pH value in the heartwood of teak.

Properties	AIAC	Ca content	Mg content	K content	Na content	pH value
Ash content	0.80**	-0.05	-0.01	0.47*	0.37	-0.26
AIAC		-0.12	0.04	0.33	0.28	-0.22
Ca content			-0.23	-0.14	-0.38	-0.12
Mg content				-0.12	-0.41*	0.79**
K content					0.72**	-0.49*
Na content						-0.55**

Note = AIAC : acid insoluble ash content * significance at the 5 % level ** significance at the 1 % level

Relationship Among the Inorganic Variables

Correlation between inorganic variables is displayed in Table 4-6. Analysis results showed a highly significant relationship between ash content and AIAC both in the heartwood and sapwood ($r = 0.71-0.80^{**}$). In the scatter diagram (Fig. 7), AIAC content obviously increased with increased ash content in a linear function. It is noted that some outliers were mostly from Kalibawang samples.

Besides silica, a significant correlation between ash content and inorganic elements was calculated on K element in the heartwood ($r = 0.47^*$). As shown in Fig. 7, K content increased polynomially when K content was greater than 100 ppm. Some outliers were distributed evenly from Kalibawang and Madiun woods. Previous work on blackening *Cryptomeria japonica* heartwood (Kubo and Ataka 1998), showed that a linear relationship between ash and K content existed ($r = 0.67$). Furthermore, it has been shown that K element was also significantly correlated with its moisture content. In this experiment, however, moisture content of the samples was not measured to confirm whether any relationships exist between inorganic elements and moisture content as trees need water to transport the nutrients.

No significant correlation among AIAC and other inorganic elements was measured. Among the elements,

the strongest degree of correlation was found between K and Na ($r = 0.72^{**}$) in the heartwood. The study showed that K content increased with increased Na content in a polynomial function (Fig. 8) although several outliers appeared. It is noted that both elements belong to the alkali metals. As the functions of K in plants is mainly for electrochemical role, it is hypothesized that univalent cations of those elements are complementary for each other. On the contrary, no significant correlation in the sapwood region was interpreted as the high mobility of main elements due to physiological activities in trees would obscure the actual concentration.

When the data in the sapwood and heartwood were combined, a significant negative correlation was found between Mg and Na ($r = -0.46^{**}$). The scatter diagram between the two displayed increased Mg content which was followed by the polynomially decreased Na content, and otherwise (Fig. 8). Mg belongs to the IIA as Na from IA groups. Mg is a mobile element and a catalyst in enzymatic reaction in physiological process (Okada *et al.* 1987). Although Mg and Na are quite different in chemical properties, this relation is interpreted as showing that they can substitute each other in some cases. The same explanation was also applied also for the negative correlation between Ca and Na to a lesser extent ($r = -0.35^*$).

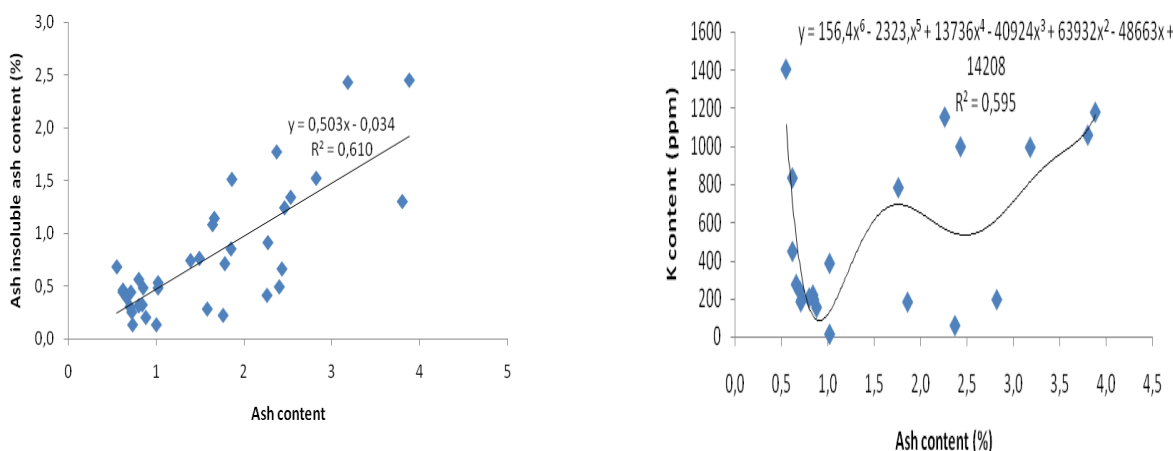


Figure 7. Scatterplots between ash content value against acid insoluble ash content in the sapwood - heartwood and between ash content against K content in the heartwood.

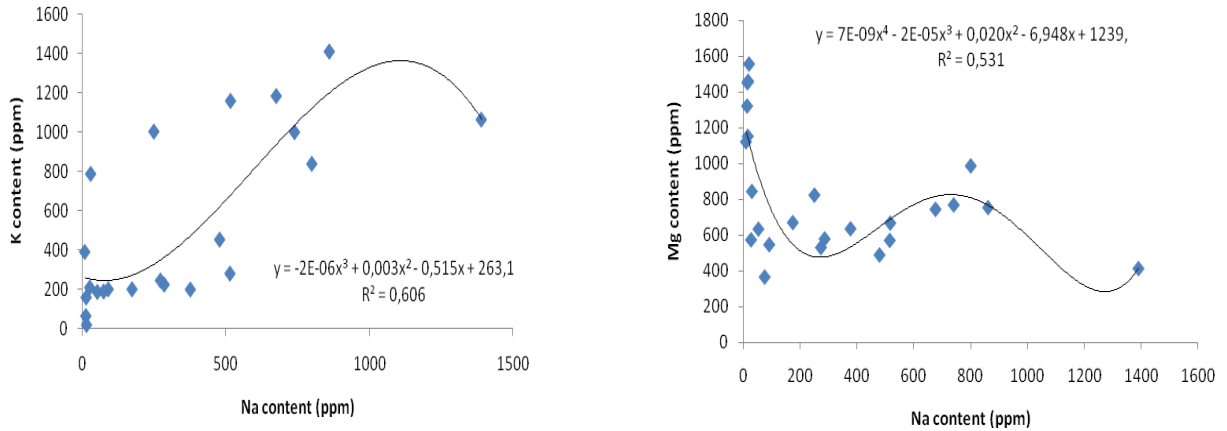


Figure 8. Scatterplots between Na content against Mg content and between Na content against K content in the heartwood.

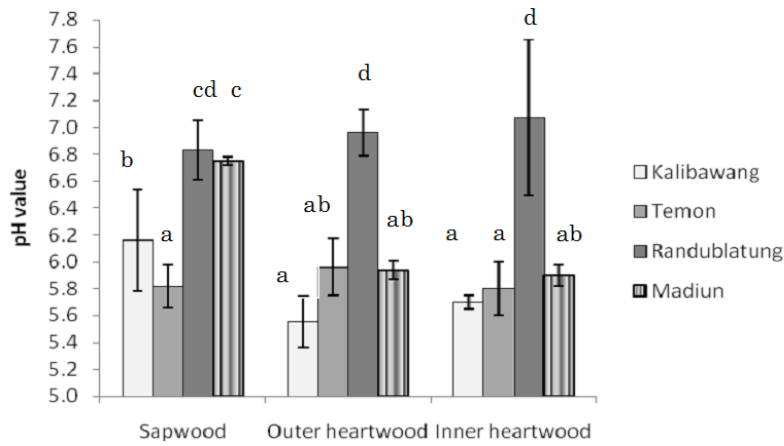


Figure 9. The pH value of teakwood by different sites and radial position. Average of 3 trees, with the standard deviation in the error bar. The same letters are not significantly different at $p < 5\%$ by Duncan's test.

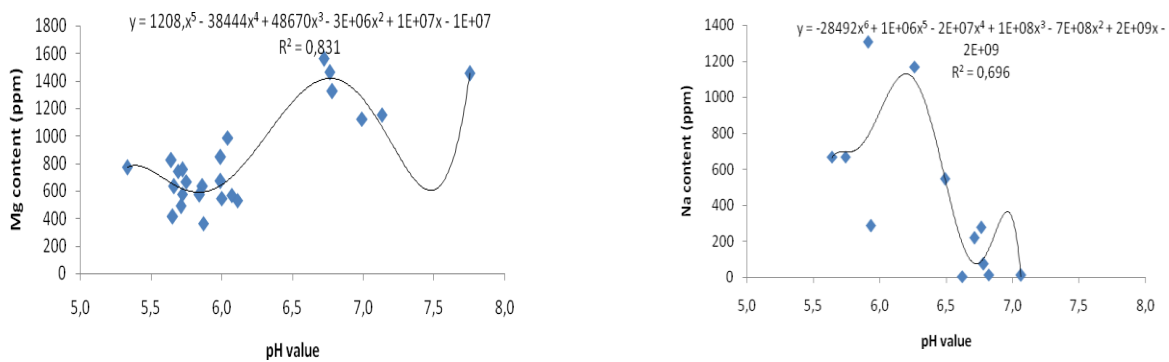


Figure 10. Scatterplots between pH value against Mg content in the heartwood and between pH value against Na content in the sapwood.

pH Values

The pH of wood meal suspensions was taken to estimate the acidity of wood. The pH values in the heartwood and sapwood were found to lie between 5.33-

7.35 and 5.64-7.06, consecutively. Those values were in the range of previous results in teakwood from Java which ranged 5.2-6.7 (Lukmandaru 2009; 2012) and teakwood from Brazil i.e. 4.6-6.7 (Polato *et al* 2005). By Duncan test,

the Randublatung woods had the highest values especially in the inner (6.96) and outer heartwood (7.07), while Temon sapwoods (5.82) and inner heartwoods (5.80) as well as Kalibawang outer (5.55) and inner heartwoods (5.70) had the lowest values. The difference between the two groups was seen in the sapwood parts of which Perhutani samples showed higher values than those of community forests (Fig. 9). Another striking difference was found in the pH values of the heartwood samples from Randublatung, which were in a weak basic range. The relationship between the values of soil pH (Table 1) and wood pH is unclear. For example the soil pH values of Temon and Randublatung were comparatively high (weak basic) but showed different trends in the pH values of the wood.

Wood acidity is derived from hydronium ions mainly released by free and bound organic acids present in extractives and non-cellulosic sugar as well as by simple phenols and complex polyphenols (Balaban and Ucar 2001). Generally, heartwoods exhibit a more acidic nature than sapwood due to the phenolics. However, this tendency was observed only in the Kalibawang and Madiun samples. Previously, the difference between sapwood and heartwood was seen in the earlier study of Panama teakwood (Windeisen *et al.* 2003). Thus, the further research should cover phenolics and short-chain sugars measurements to explain the site differences.

Wood acidity is affected by several factors, one of them being inorganic elements (Rowell *et al.* 2005). The correlation coefficients calculated between inorganic variables and pH values are shown in Table 4-6. Based on these correlations, it was clear that the levels of ash content or AIAC could not substantially explain the variation in pH values. The most correlated variable to pH values was Mg content in the sapwood and heartwood ($r = 0.65^{**}$). The degree correlation in a polynomial relationship was stronger ($r = 0.79^{**}$) in the heartwood only (Fig. 10). This trend agreed with the highest values of Mg content (1323 ppm) and pH value (6.95) in Randublatung woods (Fig. 5 and 9). This could be explained by the fact that divalent Mg^{2+} would direct the pH value to a basic range. Theoretically, Mg is easily translocated and functions as the activator of many enzymes (Okada *et al.* 1987).

On the contrary, the pH value was inversely correlated to K ($r = -0.49^*$) or Na ($r = -0.55^{**}$) contents measured in the heartwood. Okada *et al.* (1987) assumed that the high amount of alkali metals in the heartwood is to regulate the pH as a counter ions of phenolics formation. In the sapwood, a moderate and negative correlation between the pH value and Na content ($r = -0.67^*$) was measured. This means that increased Na content was followed by decreased pH values or in other words, towards the acidic range. The scatter diagram showed a polynomial relation between the two (Fig. 10). It was found that several outliers were from Temon and Randublatung woods. This association was unexpected as Na belongs to the alkali metal (IA) and directs to basic pH by carbonates neutralization (Balaban and Yilgor 1995).

Furthermore, phenolics are still limited as the heartwood has not been formed. The cause of this phenomenon is uncertain. Another factor could be the such as the presence of non-cellulosic sugars in the sapwood region.

Conclusions

Ash content and pH value were dependent on the interaction of site and radial position. The highest ash content was measured in the heartwood samples of Kalibawang whereas the Randublatung woods had the highest pH values in the heartwood regions. In comparison of samples of different sites, the woods from the Perhutani plantation (Madiun and Randublatung) was significantly richer in sodium than the woods from Kalibawang and Temon. Kalibawang teak wood contained more acid insoluble ash and potassium than others. The position of the wood in relation to radial position was another source of variability. There were larger amounts of divalent cations (magnesium, calcium) in the Perhutani plantation stands, whereas monovalent alkalines (sodium, potassium) were more abundant in the Kulon Progo stands. The radial gradient of potassium content was characterized by higher levels in the sapwood than the heartwood. There were larger amounts of acid insoluble ash content in the outer heartwood parts. The concentrations of all metals tested showed no significant differences in the heartwood region. Variation in the soil properties, in general, could not adequately explain the variation in wood properties in this experiment. Significant correlations were found between ash content and its fractions as well as between inorganic variables and pH values.

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Properties of Included Phloem in Teakwood

Ganis Lukmandaru

Abstract

In some areas of Indonesia, the heartwood of teak tree (*Tectona grandis* L.f.) contains included phloem, which is categorized as defects. This paper characterized the colour and chemical properties of such abnormal wood. Three selected trees from Perhutani plantation, Randublatung region, were assessed. The heartwood colour properties were measured by CIELAB system. Result showed that the included phloem-containing heartwood (IPHW) was darker (L^*), but less red (a^*) and yellow (b^*) compared to the adjacent normal heartwood (NHW). The lignin and ash contents were not significantly different in the wood radial direction. In contrast, the level of extractive contents were significantly different between sapwood and heartwood. The amount of ethanol-benzene extractive and solubility in 1% NaOH in the IPHW region were significantly higher than that in normal tissues. The analysis of extractive components using gas chromatography-mass spectrometry showed that the deoxylapachol and lapachol was highly marked in the IPWH region. The obtained results suggest that naphthaquinone compounds were related to the protection against wood-destroying organisme attack.

Keywords: *Tectona grandis* L.f, included phloem, chemical properties, colour properties, extractives.

Introduction

Teak is undoubtedly the main commodities of hardwood in the world for its advantageous properties and its silvicultural aspect. In Indonesia, teak is mainly planted in the Java Island and become naturalized along the time. Supply of teak timber for industries in Indonesia has been mainly provided by the state company, i.e. Perhutani Enterprise. However, cultivation of teak has been intensified by community forests to meet the high demand in the last decades.

Superiority of teak wood is due to its natural durability, strength, weather resistance as well as its beautiful grain and colour. However, in the field, such as in Perhutani forest, there are some abnormalities or defects caused by nature on the teak timber. One of them is included phloem or phloem tissue lying within the secondary xylem. The included phloem is categorized as defects based on the Indonesian National Standard of log or sawn-wood (BSN 2010a; 2010b). The frequent appearances of included phloem in the transverse surface of the wood could indicate the strength properties of the wood. The more frequent it occurs, the less strength wood does (Lee and Shum 1962; Chow *et al.* 1990). Also, it causes the inhomogenous appearances in the wood surface, such as the appearance of a darker heartwood colour compared to the normal ones.

The included phloem phenomenon appears also in several woody species, such as *Calycopteris floribunda* (Rajput *et al.* 2009), *Combretum erythrophyllum* and *Strychnos madagascariensis* (Carlquist 2013). However, there is not much information available in describing the existence of included phloem in teak and its frequency in the teak plantation. Based on our limited observation in the

fields, the included phloem in the teak stem wood occurs along with the attacks of *Neotermes tectonae*, a dry-wood termite. The attack of *Neotermes tectonae* itself, which is called 'inger-inger' or 'gembol' in Indonesian, is marked by the tumorous-shape appearances in the stem. Thus, the appearance of included phloem formation in teak has indicated a plant defense mechanism against termites.

Previous studies about the included phloem in several species were more focused to the wood formation and its anatomical properties (Outer and Veenendaal 1995; Rajput *et al.* 2008; Rajput *et al.* 2009; Veenendaal and Outer 1993). One of the studies has addressed the decay resistance of the included phloem in *Koompassia excelsa* wood and has compared it to the normal tissues (Wong 1988). Our previous works have explored the chemical characterization of abnormal wood in teak (Lukmandaru *et al.* 2009; Lukmandaru 2011; Lukmandaru 2015). Therefore, this study investigated the colour and chemical properties of the included phloem stem wood in radial direction.

Materials and Methods

Sample Collection and Preparation

Samples of teak wood were harvested from three trees (class age of 41 to 50 years) grown in Perhutani plantation, Randublatung Region, Central Java Province. Those trees were marked by the attacked of *Neotermes tectonae* termites and the appearance of thin black streaks around the annual ring. The included phloem-contained samples were selected. The wood blocks were sawn from trees in 5 cm thicknesses. Those blocks were selected from the middle part (4 to 7 m) of the trees. They were carefully

divided into three different zones from outside to inside (toward the pith): sapwood (SW), included phloem containing heartwood (IPHW) and the adjacent normal heartwood (NHW) as shown in Fig. 1. The wood samples of each region were ground to size 40-60 mesh (0.4 mm) sieve in order to obtain homogeneous particle sizes and to remove fine particles. This wood meal was used for analysis of colour and chemical properties.

Colour Measurement

The colour of the specimens was determined from the scanned specimens and expressed in CIE L* a* b* system by NF777 spectrophotometer (Nippon Denshoku). The -absorbance read at the wavelength interval of 400-700 nm. The illuminant A type (tungsten halogen light) was used and a 10° standard observer was employed. The aperture of the sensor head was 6 mm. The brightness of the colour was represented by L*-axis, while the chromaticity coordinates were represented by a*-axis and b*-axis. In the CIE L* a* b* coordinates, + a* stands for red, - a* for green, + b* for yellow, - b* for blue, and L* varies from 100 (white) to zero (black). All measurements were carried out in triplicate.

Chemical Analyses

Chemical properties, such as ash content, 1% NaOH, hot-water, cold-water and ethanol-benzene solubility were determined according to ASTM (1984a; 1984b; 1984c). Klason lignin values (TAPPI 1992) were also determined. All assays were carried out in duplicate.

Gas Chromatography-Mass Spectrometry Analyses

The extracts of ethanol-benzene were dissolved in concentration of 25 mg/mL. The GC-MS analyses were carried out using an Shimadzu QP500 fitted with a NB-1 capillary column (30 m, 0.25 mm i.d.; 0.25 µm film thickness). GC: oven temperature was programmed from 120 to 300°C rising with a 4°C/min rate and held isothermally at 300°C for 5 min. Injector temperature was 250°C. The carrier gas was helium at a 1 mL/min flow. The injection volume was 1 µL with a split ratio 1:80. EI-MS: the electron energy was 70 eV. Ion source and the connection parts temperature were 250°C. Constituents were identified by comparing experimental retention indices with those of reference compounds (2-methyl anthraquinone, lapachol, 2-hydroxymethyl anthraquinone, squalene, palmitic acid) run under identical conditions and literature data (Lemos *et al.* 1999; Perry *et al.* 1991; Windeisen *et al.* 2003). For the quantification, 2-methyl anthraquinone (tectoquinone) was used as internal standard. The concentration of each

component is expressed as mg/g of the total amount of oven dry wood powder.



Figure 1. Sampling position on a cross section of heartwood containing included phloem in teak from three trees. Remarks: SW = sapwood, IPHW = included phloem-containing heartwood, NHW = normal heartwood.

Statistical Analyses

The data were statistically calculated by one-way analysis of variance (ANOVA) and Duncan's post hoc test with $p < 0.05$ was considered to be statistically significant. Statistical analyses were done by means of SPSS 16 under Windows.

Results and Discussion

Colour Measurement

The colour of wood powder samples in radial direction was measured and the results are described in Fig. 2. The ranges for brightness (L*), redness (a*), and yellowness (b*) in the sapwood region were 51-61, 9-11 and 21-24, whereas the ranges in the heartwood were 32-45, 8-15, and 15-24, respectively. All colour measurements were significantly affected ($p > 0.01$) by radial direction. The IPHW region was significantly darker than the NHW region (L* less than 3-7 units), although the a* and b* levels in IPHW were about 3-4 units lower than the NHW. The a* levels between the sapwood and heartwood regions were not significantly different. On the other hand, the L* value of heartwood was lower to 9-10 units than the value in sapwood.

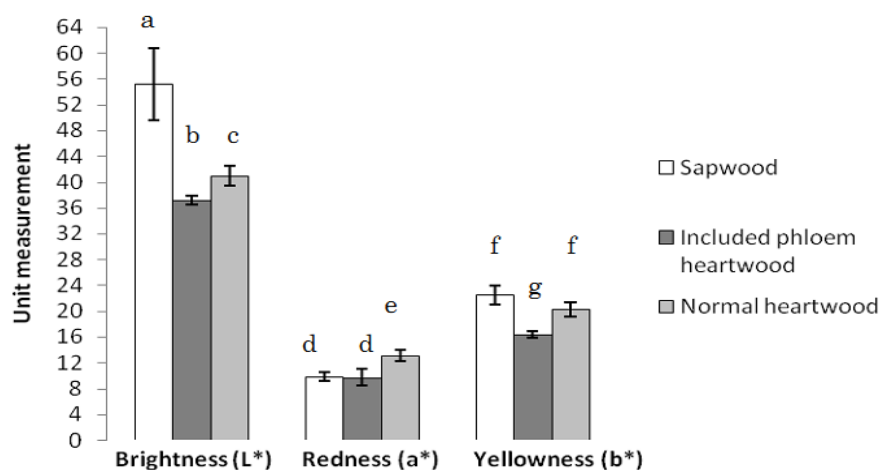


Figure 2. Colour properties in L* (brightness), a* (redness) and b* (yellowness) of teakwood containing included phloem by radial position. Mean of three trees with error bar as standard deviations. The same letters on the same graphic are not statistically different at $p < 0.05$ by Duncan's test.

Based on the field observation at the same location, the stem with black-streak frequently occurs without the included phloem. Previous findings showed a lesser value of brightness (L*) to 12-15 units and yellowness (b*) to 3-4 units in the black streak part (Lukmandaru *et al.* 2009). This raised question whether the occurrence of included phloem is related to the black-streak phenomenon in teak. Measurement of the L* values in three replicates of trees could not firmly conclude the effect of blackening as shown by the difference of merely 3 units value. To assess the effect of blackening, the heartwood samples with included phloem but without the black-streaked are necessary to study at the same site.

Chemical Analyses

Measurements of extractive and ash contents were displayed in Fig. 3. The range of ethanol-benzene soluble extractive, hot-water solubility and cold-water solubility contents in the sapwood were 4.73-6.72%, 2.86-3.92%, and 5.78-7.98%, whereas the ranges in the heartwood were 5.78-20.16%, 0.85-2.16% and 0.98-2.43%, respectively. The ANOVA revealed that the effect of radial direction was highly significant to ethanol-benzene soluble extractive and cold-water solubility contents ($p > 0.01$) and was significant to hot-water solubility contents ($p = 0.01$).

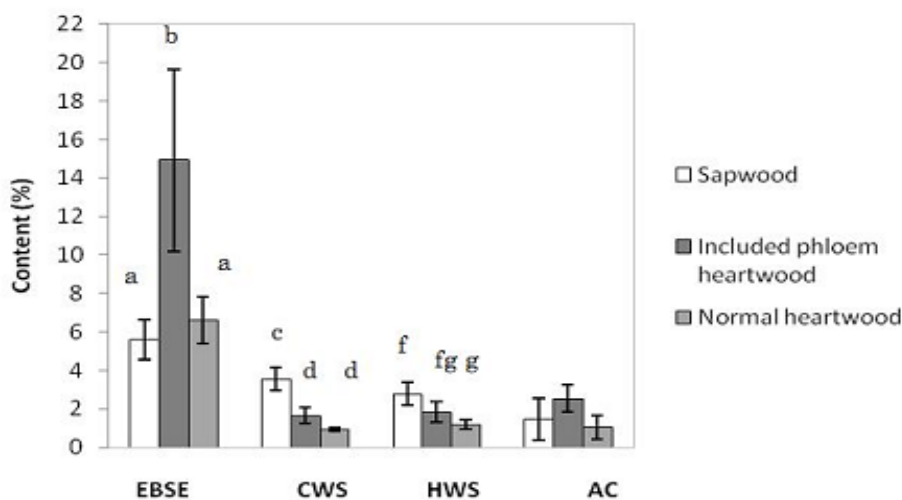


Figure 3. The extractive and ash contents of teakwood containing included phloem by radial position. Mean of three trees with error bar as standard deviation. The same letters on the same graphic are not statistically different at $p < 0.05$ by Duncan's test. EBSE : ethanol-benzene soluble extractive content, CWS = cold-water solubility content, HWS = hot-water solubility content, AC = ash content.

The ethanol-benzene soluble extractive amounts were significantly different ($p < 0.05$) between the IPHW and NHW. The mean of ethanol-benzene soluble extractive content in the IPHW was 14.90% compared to 6.69% in the NHW. This is possibly due to the higher extractive contents of the phloem (bark) mixed in the wood. The comparatively high content of ethanol-benzene soluble extractive in the IPHW was not observed in the amounts of cold-water and hot-water solubility. It is an unexpected finding as barks normally has a higher water solubility content than the woods (Fengel and Wegener 1989). The extractive contents by successive extraction were similarly found in the black-streaked heartwood of teak (Lukmandaru *et al.* 2009). Thus, contribution of blackening phenomenon to the colour properties of the black-streak heartwood is remain unknown. The high levels of cold-water and hot-water solubility in sapwood regions indicate the high content of primary extractives such as sugars.

The range of ash content in the sapwood and heartwood was 0.54-2.68% and 0.48-3.33%, respectively. Radial direction has no significant effect ($p = 0.15$) in this matter. Similar to the cold-water and hot-water solubility, the comparatively high ash content levels in the sapwood is due to its physiological function, i.e. absorbing the nutrients from the soil. There is no significant difference in ash contents between sapwood and heartwood, which may probably due to the presence of phloem in the heartwood. In general, barks posses a higher ash content compared to woods (Sjostrom 1993). However, it could not adequately explain the insignificancy between the IPHW and NHW values.

The lignin content and 1% NaOH solubility are presented in Fig. 4. The lignin content of the sapwood and

heartwood regions ranged from 35.1-37.0% and 34.8-37.6%, whereas, the 1% NaOH solubility contents ranged from 19.43-24.12% and 12.90-28.24%, respectively. Radial direction was not significantly affect lignin content ($p = 0.57$). However, it was significantly affect 1% NaOH solubility content ($p = 0.04$). Lignin is known as a protection component against microbial wood degradation (Henriksson 2009). The insignificant of lignin content indicates the presence of included phloems which was not affected the composition of cell wall component. The level of NaOH solubility in the IPHW (24.62%) was significantly higher than the level in the NHW (16.77%). Previous studies have reported the high content of 1% NaOH solubility in the barks (Kofujita *et al.* 1999; Usta and Kara 1997; Voulgaridis *et al.* 1985). This result indicates the presence of higher amount of low-molecular weight sugar in the IPHW region, as the short-chain sugars could be derived from its phloem (Hillis 1987).

Gas Chromatography-Mass Spectrometry Analysis

GC-MS analysis of ethanol-benzene extracts are reported in Fig. 5. The chromatograms presented about 10 main peaks. Identification of the products using standard components and literatures indicated the presence of quinones (lapachol, deoxylapachol and its isomer, tectoquinone and tectol), palmitic acid and the dominants triterpene, squalene, along with two unidentified compounds (Un1 and Un2). Those identified compounds were also detected in previous reports (Lukmandaru 2012; Lukmandaru and Takahashi 2009; Niamké *et al.* 2011; Windeisen *et al.* 2003).

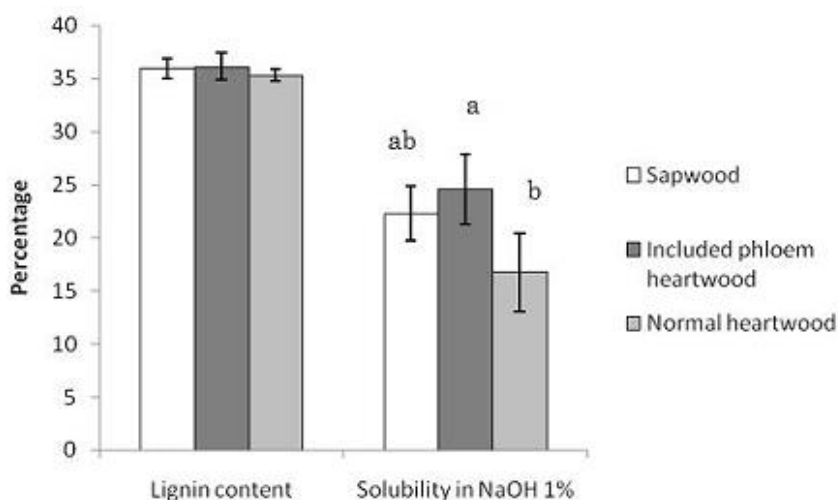


Figure 4. The lignin content and solubility in 1 % NaOH of teakwood containing included phloem by radial position. Mean of three trees with error bar as standard deviation. The same letters on the same graphic are not statistically different at $p < 0.05$ by Duncan's test.

The quantification of major compounds is summarized in Table 1. There was a statistically significant difference in deoxylapachol and its isomer, lapachol, Un1 and squalene. The amounts of deoxylapachol and its isomer, lapachol and Un1 were significantly higher in the IPHW region than those in the NH. While, the squalene level in the sapwood was significantly different to the level in heartwood. Chemical structures analysis has characterised lapachol, deoxylapachol and its isomer as naphtaquinone, while tectoquinone as an anthraquinone and tectol as a dimeric naphtaquinone. The molecular masses of unidentified compound (Un1) was found to be m/e (base peak) for 244.

The formation of included phloem could be a kind of protection of phloem against insects and other pests attacks in one to several layers of wood (Mauseth 2014). The natural decay resistance of included phloem was higher than that of normal tissues in *Koompassia excelsa* wood as reported in Wong (1988). Previous reports demonstrated that deoxylapachol effectively inhibited the growth of some fungi (Sumthong *et al.* 2006, 2008; Lukmandaru 2013). Thus, the formation of included phloem might be related to the *Neotermes tectonae* termite attacks (Lukmandaru 2015).

However, one study of termiticidal activities showed that both lapachol and deoxylapachol were not as effective as tectoquinone (Sandermann and Simatupang 1966). In this present experiment, the amount of tectoquinone in the IPHW and NHW was not significantly different. This result is contrast to the previous report of black-streak parts of teak heartwood which contains high tectoquinone amounts (Lukmandaru *et al.* 2009). It is due to the wide variations in the amount of tectoquinone and other major constituents found in different trees by examining standard deviations (Table 1).

Deoxylapachol is hypothesized as the precursor of both tectoquinone and tectol (Sandermann and Simatupang 1966). Comparatively high level of the naphtaquinone derivatives in the IPHW has been interpreted as a defense mechanism by slowing the conversion of naphtaquinone into more complex components. Future studies with larger number of tree samples and different locations, as well as further bioassay testing and characterization of unknown compound (Un1) would be beneficial to clarify the assumptions above.

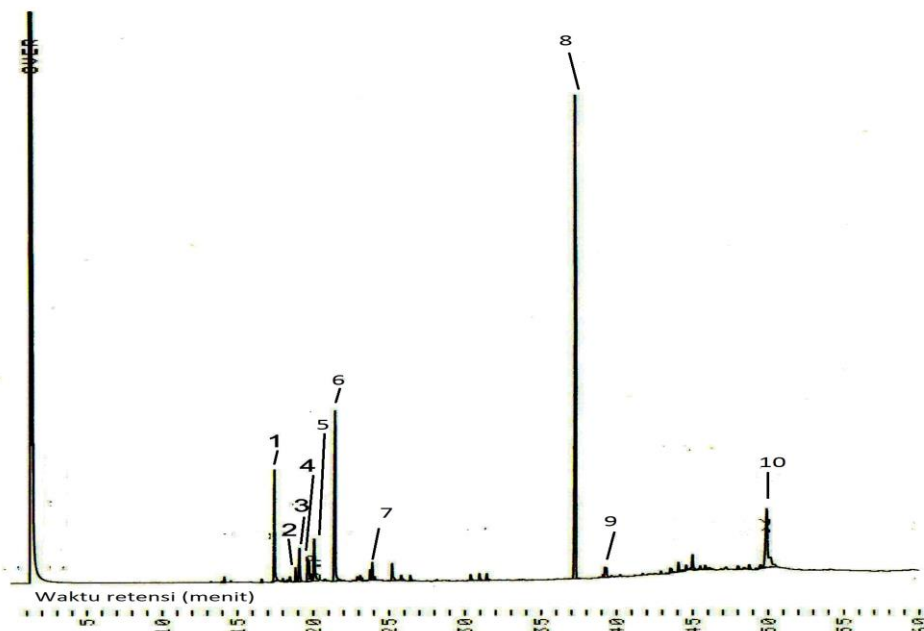


Figure 5. Gas chromatogram of the ethanol-benzene extracts from heartwood containing included phloem in teak (1 & 5. Deoxylapachol or its isomer; 2. Palmitic acid; 3. Unknown; 4. Lapachol; 6. Tectoquinone; 7. Unknown; 8. Squalene; 9. Unknown; 10. Tectol).

Table 1. The content of extractive components (mg/g oven dry wood) in teakwood containing included phloem by radial position.

No.	Component	GC-MS analysis		Radial position		
		Ret. time (minutes)	Sapwood	Included phloem - heartwood	Normal heartwood	Sig.
1.	Deoxylapachol	17.41	5.46 (4.90)a	23.2 (7.92)b	0.15 (0.08)a	0.01>**
2.	Palmitic acid	18.62	0.03 (0.05)	0.33 (0.30)	0.23 (0.15)	0.25
3.	Un1	18.93	0.36 (0.46)a	2.03 (0.92)b	0.23 (0.15)a	0.01*
4.	Lapachol	19.48	0.73 (0.25)a	7.73 (5.68)b	1.30 (0.36)a	0.04*
5.	Isodeoxylapachol	19.84	3.73 (4.02)a	11.20 (4.61)b	1.76 (0.40)a	0.03*
6.	Tectoquinone	21.17	1.83 (1.10)	25.56 (23.18)	7.10 (0.95)	0.15
7.	Un2	23.65	2.60 (3.89)	2.20 (1.70)	0.66 (0.25)	0.62
8.	Squalene	37.27	6.60 (3.37)a	16.40 (9.60)b	26.53 (11.70)b	0.04*
9.	Un3	39.35	0.80 (0.80)	1.10 (1.15)	2.66 (4.35)	0.66
10.	Tectol	50.06	3.36 (3.25)	3.46 (2.97)	4.90 (1.56)	0.74

Remarks: Mean of three trees with standard deviation in parentheses. The same letters on the same row are not statistically different at $p < 0.05$ by Duncan's test.

Conclusions

The characteristics of the included phloem-containing heartwood (IPHW) parts of teak were examined for colour and chemical properties. IPHW showed a darker but less red and yellow colour than the adjacent normal heartwood. The IPHW portion also contained ethanol-benzene extractives. Its 1% NaOH solubility contents was significantly higher than the content of normal heartwood parts. Between the sapwood and the IPWH, significant differences were observed in the ethanol-benzene and cold-water soluble extractives. Lignin contents in radial direction did not vary significantly. Among the extractive components, the lapachol, deoxylapachol and its isomer, as well as the content of unknown compound (Un1) were particularly high in the IPWH portion, which suggests some relation of naphthaquinones with the defense mechanism of teak.

Acknowledgements

The author is gratefully acknowledged Prof. Koetsu Takahashi (Faculty of Agriculture, Yamagata University, Japan) for facilitating this research. Author is also thankful to Mr. Untoro Tri Kurniawan (Perhutani Enterprise) for providing the samples.

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Wood Anatomical Features and Physical Properties of Fast Growing Red Meranti from Line Planting at Natural Forest of Central Kalimantan

Joko Sulisty, Harry Praptoyo, Ganis Lukmandaru, Ragil Widyorini, Widyatno, Oka Karyanto, Futoshi Ishiguri, and Sri Nugroho Marsoem

Abstract

High productivity fast growing species plantation establishment such as the line planting of red meranti (i.e. *Shorea leprosula* and *Shorea parvifolia*) with intensive silviculture is one potential solution to improve wood supply for industries in Indonesia. However, the information of anatomical properties and wood properties of these two species related to the influence of the line planting system and tree growth rate is limited. This paper studies the anatomical features, wood cell proportions, fiber dimensions and physical properties of wood in radial variation in relation to the line planting effect and tree growth rate. Wood of the trees grown in the line planting system showed higher proportion of vessel element compared to those of wood from natural forest. The vessel diameter of wood from the line planting was also larger than that of in wood from natural forest. The specific gravity of wood from *Shorea parvifolia* grown on the line planting was higher than that of wood grown in natural forest. The variation of specific gravity on wood portion near to the pith of *Shorea leprosula* and *Shorea parvifolia* trees grown on the line planting was related to the variation of the cell wall thickness. The bigger diameter of trees grown or the faster growth rate in the line of planting at the same age shows the greater vessel diameter in wood of *Shorea leprosula* and *Shorea parvifolia* and greater specific gravity of *Shorea parvifolia* wood.

Keywords: Fast growing red meranti, wood cells proportion, fiber dimension, physical properties.

Introduction

In the last decade, there was a gap of wood material supply and the capacity of wood industries in Indonesia. The wood material supply from forest is unable to meet the industries' demand. For example in 2010, the production capacity of sawmill and plywood/LVL was 13-2.7 million and 20.8-3.6 million m³/year, respectively. On the other hand, the raw material supply for these two industries were only 5.5 and 6 million m³ (Ministry of Forestry 2010). These conditions also deteriorate the sustainability of forest in Indonesia. Establishment of fast growing species plantations with high productivity such as the line planting of red meranti including *Shorea leprosula* and *Shorea parvifolia* with intensive silviculture in tropical rain forest are potential solutions of these problems (Na'iem and Widiyatno 2012). The line planting of red meranti trees species is expected to improve the productivity in tropical forest from 3 m³/ha/year to 300 m³/ha/year. The establishment of the line planting in tropical rain forest also shows another advantage i.e. to keep the high biodiversity in the area between the lines planting. Fig. 1 shows a schematic design of the line planting in tropical rain forest.

During the initial 5 years-old tree planted on the lines in tropical rain forest, there are clearing treatments for 5 times in the lines in order to open the space to make the trees planted have open space to receive enough sunlight (Na'iem and Widiyatno 2012). The availability of sunlight might be desirable for tree growth and tends to maximize the density and strength of the trees (Shmulsky and Jones 2011). The field observations are found the good

performances of red meranti trees including *Shorea leprosula* and *S. parvifolia* i.e. fast growing, straight and cylindrical stems, tall free branches stems, etc. However, there is less information of the anatomical properties and wood properties of these two species related to the influence of the line planting system and tree growth rate. This paper study the anatomical features, wood cell proportions, fiber dimensions and physical properties of wood in radial variation related to the line planting effect and tree growth rate. This study observed 10 years-old two red meranti species i.e. *S. leprosula* and *S. parvifolia* grown on the line planting with diameter variation from 7, 15 and 20 cm. These two tree species from natural forest with diameter of 20 cm was also observed.

Materials and Methods

Wood disc samples of *S. leprosula* and *S. parvifolia* were obtained from the bottom part of about 1.3 m above ground of 10 years-old trees grown on the line planting in natural forest concession managed by PT. Sari Bumi Kusuma, Central Kalimantan. Each tree species were selected from sound trees showing cylindrical and straight stem characteristics with variation on diameters i.e. 7, 15 and 20 cm. Trees of these two species grown naturally on natural forest with diameter of 20 cm were also cut for this study (Table 1). The wood disc was then cut into a strip with a thickness of 5 cm. The wood strip was cut for every 1 cm in the radial position for the observation of anatomical features, proportion of wood cells, fiber dimensions and physical properties.

Fiber dimensions were analyzed from square wood samples with the size of 1 x 1 x 20 mm (tangential, radial and longitudinal) which was taken from every 1 cm on the radial position from pith to the area close to the bark of tree. The wood samples were macerated with the mixed solution of glacial acetic acid and hydrogen peroxide (1:20) and heated at 100°C for more than 4 hours. Stained wood fibers were then placed on deck-glass. The wood fibers of each species were photographed by an optical light microscope (OLM, Olympus BX 51 DP 72, Japan). The dimension

including fiber length, cell diameter, cell wall thickness and lumen diameter, as well as proportion of dimension of wood fibers were examined and recorded. Preparation and measuring of the physical properties of wood including green moisture content and green specific gravity were carried out based on *British Standard* no 373, 1957. The wood samples of physical properties were taken from every 1 cm on the radial position from pith to the area close to the bark of tree.

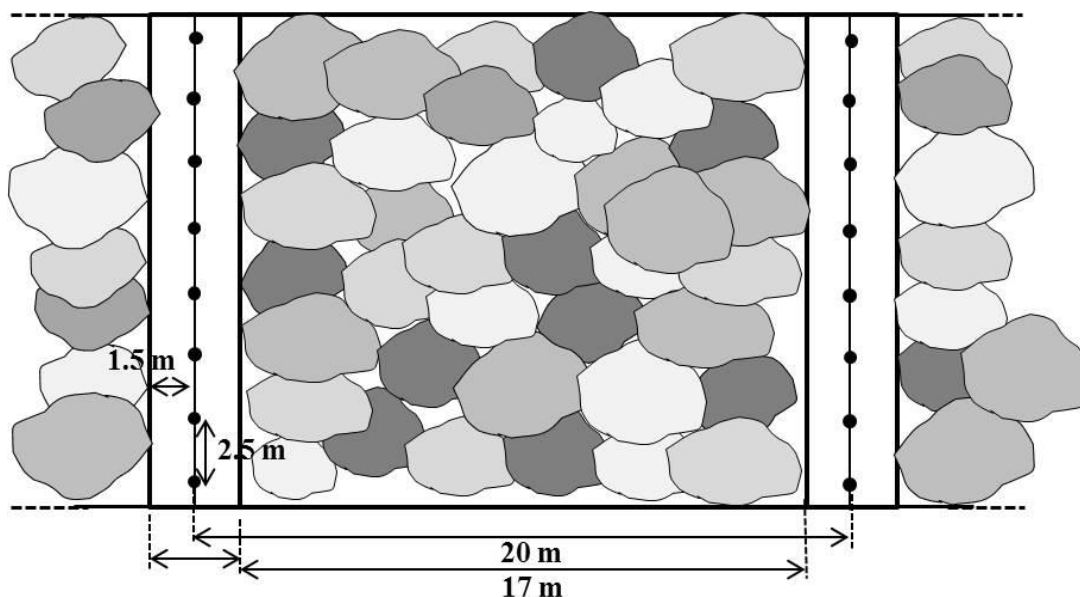


Figure 1. Schematic design of the line planting with intensive silviculture in tropical rain forest

Table 1. Two species of meranti trees were cut from the line planting and natural forest sites

Species	Age (year)	Ø (cm)	Site
<i>Shorea leprosula</i>	10	7	Line planting
	10	15	Line planting
	10	20	Line planting
	-	20	Natural forest
<i>Shorea parvifolia</i>	10	7	Line planting
	10	15	Line planting
	10	20	Line planting
	-	20	Natural forest

Results and Discussion

Fig. 2 shows the anatomical features on the cross section, radial and tangential surfaces of *S. leprosula* and *S. parvifolia* wood. Both *S. leprosula* and *S. parvifolia* wood

possessed solitary and two radial multiples pores or vessel (Martawijaya *et al.* 1989).

Fiber showed the largest proportion on both wood species of *S. leprosula* and *S. parvifolia* with the proportion in the range of 39.1-56.3% and 48.8-61.2%, respectively. The least cell proportion was found on resin canal and ray cells.

Meanwhile the proportion of vessel on *S. leprosula* and *S. parvifolia* was in the range of 8.5-11.3%, 8.14-11.7%, respectively. The line planting system increased the proportion of vessel and parenchyma, on the other hand influenced the decreasing the proportion of wood fiber, as

shown in Table 2. Moreover, the line planting which increases the tree growth rate influenced on the increase of vessel diameter as shown in Fig. 2. The relationship between tree growth rate and vessel diameter was similar to previous study (Sisi *et al.* 2010).

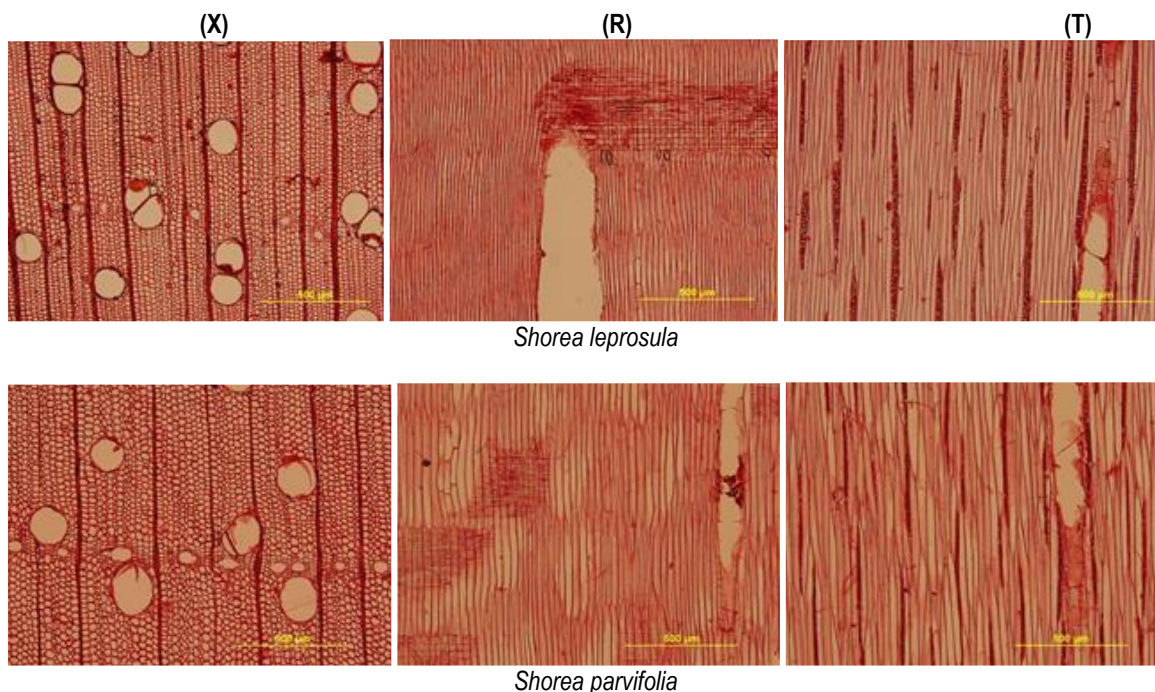


Figure 2. Anatomical features of *S. leprosula* (above) and *S. parvifolia* (below) on the cross section (X), radial (R) and tangential (T) surfaces.

Table 2. Proportion of wood cells of *S. leprosula* and *S. parvifolia* grown on the line planting and natural forest sites

Species	Site	Vessel (%)	Parenchyma (%)	Rays (%)	Fiber (%)	Resin Canal (%)
<i>Shorea leprosula</i>	Line planting	8.34	14.70	7.49	56.36	1.05
	Natural forest	7.85	13.09	8.36	56.90	1.04
<i>Shorea parvifolia</i>	Line planting	11.75	13.75	10.42	48.94	1.34
	Natural forest	8.01	11.70	8.51	59.49	1.30

Table 3 shows the fiber dimensions of *S. leprosula* and *S. parvifolia* grown both on the line planting and natural forest. The fiber length, fiber diameter and cell wall thickness of wood from *S. leprosula* grown on natural forest was higher than those of wood from the trees grown on the line planting. On the contrary, *S. parvifolia* grown in the line planting possessed the increase of fiber length compare to the fiber length from the tree grown on natural forest. The increase of fiber length on *S. parvifolia* grown on the line planting probably determined on the higher specific gravity of wood as shown in Table 4. The higher specific gravity of wood of *S. leprosula* was found from the tree grown on natural forest due to the effect of fiber length, lumen diameter and cell wall thickness. Panshin and DeZeeuw

(1980) pointed out that, in general terms, the density of wood depends on (1) the size of cells, (2) the thickness of the cell walls, and (3) the inter-relationship between those two.

Fig. 3 shows that the specific gravity of wood from *S. leprosula* and *S. parvifolia* trees grown on natural forest rapidly increased from pith to bark. The specific gravity increases from the pith to the bark has been reported by others (Huang *et al.* 2003; Jordan *et al.* 2008). The pattern of green moisture content variation on the radial position of *S. parvifolia* grown on natural forest was on the contrary with the pattern of the specific gravity variation as reported previously (Chu and Lin 2007). However, the pattern of green moisture content variation on the radial positions of *S.*

leprosula was similar to the pattern of specific gravity variation. The amount of water in cell wall or lumen of wood cell near the pith may decrease as result of deposition of extractive (Shmulsky and Jones 2011). *Shorea leprosula* and *S. parvifolia* trees grown on the line planting showed a different pattern of specific gravity of the radial positions. The specific gravity of wood near the pith of tree grown on the line planting tended to be higher than that of tree grown on the natural forest. The increase of specific gravity on part near the pith of tree grown on the line planting was probably due to the response of the clearing for 5 times in the initial 5 year to make the trees planted had open space to receive enough sunlight. The availability of sunlight might be desirable for tree growth and tends to maximize the density these trees (Shmulsky and Jones 2011).

The fiber proportion of *S. leprosula* and *S. parvifolia* showed no consistent pattern on the effect of tree growth rate or tree diameter, as shown in Table 5. The vessel proportion of *S. leprosula* and *S. parvifolia* was recognized a contrast pattern related to tree growth rate. The vessel proportion of *S. leprosula* decreased with the increase of

tree diameter or growth rate, which was similar to that *Populus nigra* (Sisi *et al.* 2010; Phelps *et al.* 1982) and *Shorea acuminatissima* (Ishiguri *et al.* 2012). In contrast, the increase of vessel proportion was found with the increase of *S. parvifolia* tree diameter or growth rate (Ishiguri *et al.* 2012).

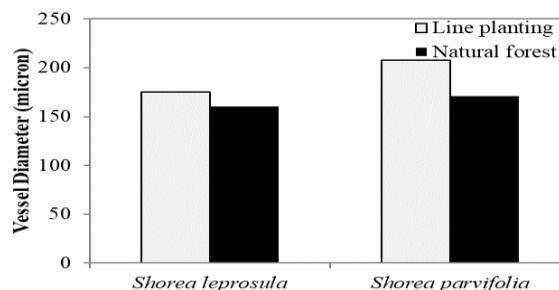


Figure 3. Vessel diameter of *S. leprosula* and *S. parvifolia* grown on the line planting and natural forest sites.

Table 3. Fiber dimensions of *S. leprosula* and *S. parvifolia* grown on the line planting and natural forest sites.

Species	Sites	Fiber Length (mm)	Fiber Diameter (micron)	Lumen Diameter (micron)	Cell wall Thickness (micron)
<i>Shorea leprosula</i>	Line Planting	1.042	22.68	19.04	1.820
	Natural Forest	1.081	22.88	18.84	2.020
<i>Shorea parvifolia</i>	Line Planting	1.095	21.65	17.94	1.851
	Natural Forest	0.955	27.16	23.15	2.005

Table 4. Green specific gravity (SG) and green moisture content (MC) of wood of *S. leprosula* and *S. parvifolia* grown on the line planting and natural forest sites

Species	Sites	Green SG	Green MC (%)
<i>Shorea leprosula</i>	Line Planting	0.255	101.6
	Natural Forest	0.277	78.1
<i>Shorea parvifolia</i>	Line Planting	0.320	118.5
	Natural Forest	0.260	97.6

Table 5. Proportion of wood cells of *S. leprosula* and *S. parvifolia* grown with different rates on the line planting

Species	Diameter (cm)	Vessel (%)	Parenchyma (%)	Rays (%)	Fiber (%)	Resin Canal (%)
<i>Shorea leprosula</i>	7	11.30	12.64	10.54	51.41	
	15	10.47	13.57	14.68	39.17	0.95
	20	8.34	14.70	7.49	56.36	1.05
<i>Shorea parvifolia</i>	7	8.14	14.74	8.89	48.79	0.88
	15	9.46	12.18	11.39	52.46	1.54
	20	11.75	13.75	10.42	48.94	1.34

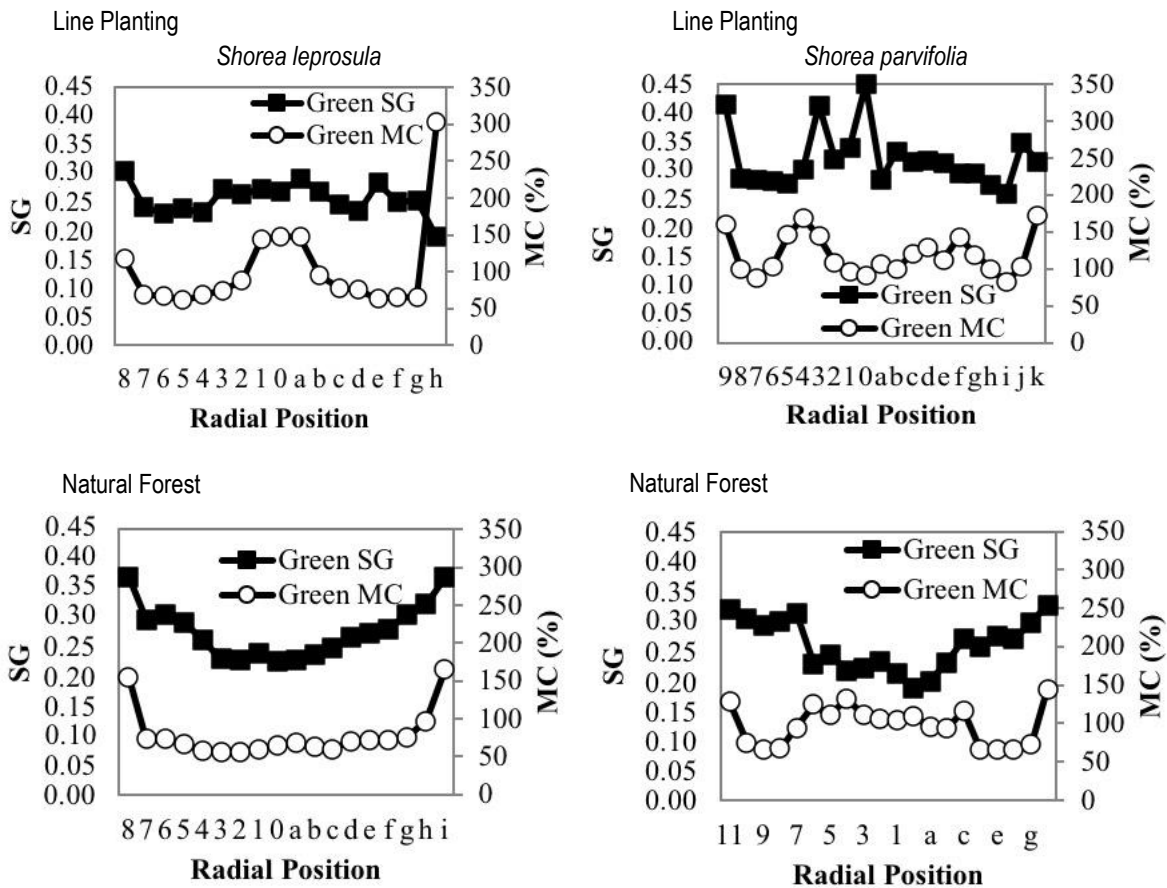


Figure 4. Variation of green SG and green MC in radial positions of wood of *S. leprosula* (left) and *S. parvifolia* (right) grown on the line planting (upper) and natural forest (below) sites.

Table 6. Green specific gravity (SG) and fiber dimensions of wood of *S. leprosula* and *S. parvifolia* grown with different rates on the line planting

Species	Tree Diameter (cm)	Green SG	Fiber Length (mikron)	Fiber Diameter (mikron)	Lumen Diameter (mikron)	Cell wall Thickness (mikron)
<i>Shorea leprosula</i>	7	0.40	1.023	22.28	17.79	2.251
	15	0.41	1.062	20.08	15.63	2.226
	20	0.26	1.042	22.68	19.04	1.820
<i>Shorea parvifolia</i>	7	0.26	0.941	23.40	19.58	1.908
	15	0.28	0.999	23.41	19.66	1.875
	20	0.31	1.095	21.65	17.94	1.851

Fig. 4 shows that the vessel diameter increased with the increase of both *S. leprosula* and *S. parvifolia* tree diameter or growth rate. These results are comparable to that found by other research on that *Populus nigra* (Sisi et al. 2010) and *Shorea acuminatissima* (Ishiguri et al. 2012).

Table 6 shows the proportion of wood cells of 10 year-old *S. leprosula* and *S. parvifolia* grown on the line planting

with different rates. The fiber length tended to increase with the increase of tree diameter from 7 to 15 cm, on the other

hand the cell wall thickness decreased. The further increase of tree diameter tended to decrease the fiber length and cell wall thickness. The interesting finding was shown by the fiber dimensions of *S. parvifolia* related the tree growth. The fiber length increase, on the contrary the fiber diameter and

cell wall thickness decreased with the increase of tree diameter of *S. parvifolia*. Therefore the increase of tree growth rate on *S. parvifolia* trees and the increase up to a certain tree diameter of *S. leprosula* increased the fiber length but decreased the fiber diameter and cell wall thickness which was determined by the increase of the specific gravity of wood (Panshin and De Zeeuw 1980). The similar result that showed the increase of basic density of wood with the increase of growth rate of *Shorea acuminatissima* trees reported previously (Ishiguri *et al.* 2012).

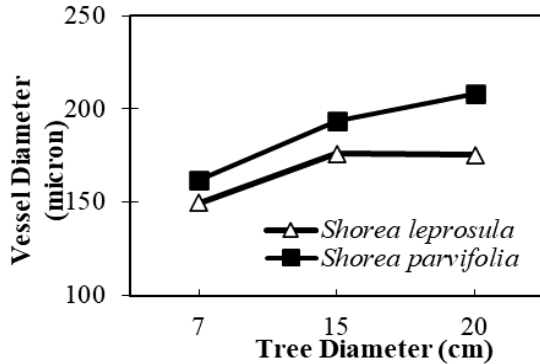


Figure 5. Vessel diameter in wood of 10 year old *S. leprosula* and *S. parvifolia* trees grown with different rates on the line planting.

Fig. 5 shows the different radial variation found for green moisture content, which was high near the pith and then decrease toward to the bark of *S. leprosula* trees. On the other hand, the green moisture content was low near the pith and then increased toward the bark of *S. parvifolia* trees. The variation of green moisture content was reported previously (Chu and Lin 2007). Tree diameter of both tree species influenced the green moisture content in the radial position. The green moisture content of tree with diameter of 7 cm possessed the thickest cell wall which was lower than those of tree with bigger diameter.

Fig. 6 shows the variation of specific gravity, fiber length and cell wall thickness on the radial positions of 10 year-old *S. leprosula* and *S. parvifolia* trees grown on the line planting with different rates. The variation of specific gravity on the radial positions of both tree species was discussed on the previous part. The effect of tree diameter on the variation of specific gravity was strongly related to the fiber dimensions as discussed on the previous part. The specific gravity variation on the radial positions near to the pith was obviously determined by the variation of cell wall thickness due to the response of the clearing for 5 times in the initial 5 year to make the trees planted had open space to receive enough sunlight. The availability of sunlight might tend to maximize the cell wall thickness which was determined by specific gravity.

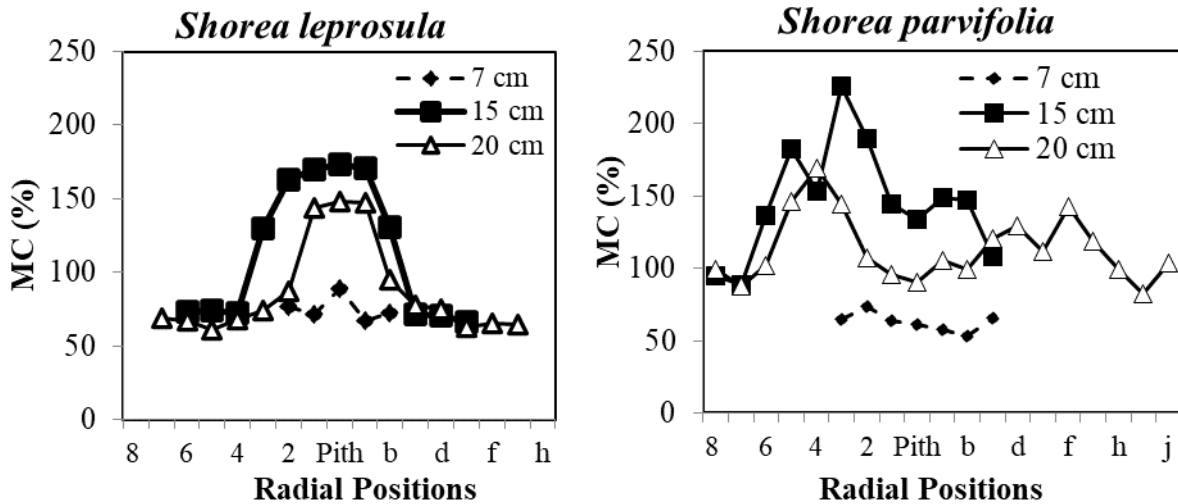


Figure 6. Variation of green moisture content (MC) in the radial positions of wood of *S. leprosula* (left) and *S. parvifolia* (right) trees grown with different rates on the line planting.

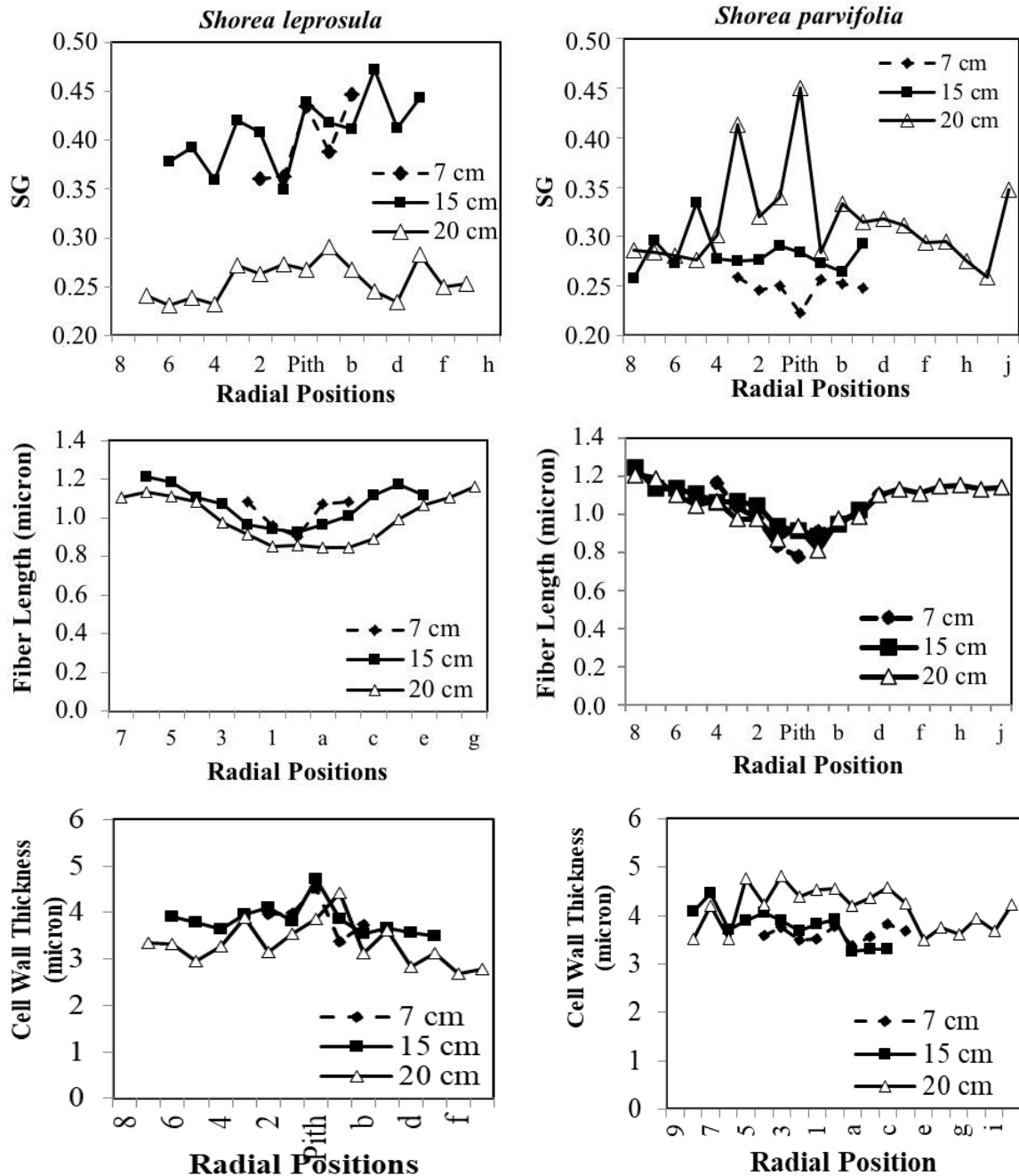


Figure 7. Variation of green specific gravity (SG), fiber length and cell wall thickness in the radial positions of wood of *S. leprosula* (left) and *S. parvifolia* (right) trees grown with different rates on the line planting.

Conclusions

The line planting system influenced on the higher proportion of vessel element in wood compare to those of wood collected from natural forest. The vessel diameter of wood from the line planting was greater than that of wood from natural forest. The specific gravity of wood

from *Shorea parvifolia* grown on the line planting was greater than that of trees grown in the natural forest. The variation of specific gravity of wood in the radial positions grown on the line planting was different to that of trees grown in the natural forest. The variation of specific gravity on part near to the pith of *Shorea leprosula* and *Shorea parvifolia* trees grown on the line planting related to the variation of the cell wall thickness. The bigger

diameter of trees grown or the faster growth rate in the line planting at the same age shows the greater vessel diameter in wood of *Shorea leprosula* and *Shorea parvifolia* and the higher specific gravity of wood of *Shorea parvifolia*.

Acknowledgements

This research was carried out with support from IM-HERE Project Faculty of Forestry - UGM (001/Act 2.3/FKT/I-MHERE B 2c/Kt/2010). The authors express sincere thanks to PT. Sari Bumi Kusuma Indonesia for providing sample trees.

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Accelerated Weathering Performances of Furfurylated and Acetylated Bamboo Sheets

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Abstract

Bamboo material is a potential wood substitute given that its physical and mechanical properties are comparable with those of wood. As lignocellulose material, bamboo is also degraded for use outdoor. Two significant chemical modification for wood which may work for bamboo material are acetylation and furfurylation. This paper evaluates the weathering performance of furfurylated and acetylated bamboo sheets. Parameters studied include colour changes and contact angle after accelerated weathering process at QUV chamber. The result shows that the total colour differences (ΔE^*) of furfurylation is higher than non-modified strips, while colour differences of acetylated bamboo strips are less than non-modified strips. To summarize, chemically modified sheets turn grey after weathering. Slowing of lignin photo-degradation by acetylation is attributed to the acetyl groups, which limits the degradation of lignin. Treating bamboo sheets with acetic anhydride and furfuryl alcohol was found to be effective in protecting bamboo from absorbing water during weather exposure.

Keywords: Bamboo sheets, acetylation, furfurylation, accelerated weathering, colour, contact angle.

Introduction

Bamboo is a woody, valuable and robust material. It grows naturally on all continents except Europe (Liese 1987) and shows potential as a wood substitute given that its physical and mechanical properties are comparable with those of wood. The most significant advantage of bamboo is its growth rate, where bamboo grows up to 30 meters within six months in some tropical countries, demonstrating the potential of substituting bamboo for slower-growing wood species to increase annual yield (Liese 1987). Additionally, the extensive root network makes bamboo a stable carbon fixate, erosion controller and water table preserver. Bamboo is an essential means of establishing reforestation and often has a positive effect on groundwater levels and soil improvement via the nutrients in its debris (van der Lugt *et al.* 2009).

Bamboo species are of enormous importance to rural people in several regions of Asia. For many centuries bamboo has played an essential role in the daily life of the people of tropical countries (Sharma 1980; Jifan 1985). Traditionally it is used for light building materials, scaffolding, ladders, mats, baskets, containers, tool handles, pipes, fencing, handicrafts, toys and musical instruments. In addition to traditional applications, modern processing techniques have considerably extended its usefulness in applications such as ply bamboo, bamboo mat board and laminated bamboo for flooring (Recht and Wetterwald 1992; Nugroho and Ando 2001).

Anatomically, the culm wall structure consists of vascular bundles which are embedded in ground parenchyma tissues. Vascular bundles are not uniformly distributed inside culms. Numerous smaller bundles are present towards the outer portions, while larger but fewer bundles are found towards the central region of the culms

(Kumar and Dobriyal 1992). The outer part, which is protected by hard kitin bark is more resistant to outdoor exposure than the inner part of the bamboo, is less resistant than those of the outer part. Exposing material to outdoor conditions is not only placing material in direct contact with solar radiation but also to rapid changes of moisture (Feist 1983). Solar radiation that contains ultraviolet light initiates photochemical degradation, which is identified through colour changes in the material (Chang *et al.* 1982). Water sorption during outdoor exposure causes uneven shrinkage and swelling that initiates material degradation. Outdoor exposure to inner part bamboo has not been studied intensively.

Currently, environmental concerns regarding the use of chemicals for wood preservation has generated interest in alternatives method such as chemical modification (Hill 2006). Wood modification is a means of altering the material to overcome one or more of its disadvantages. Two major chemical modification processes for wood modification are acetylation and furfurylation (Jones 2007). Acetylation replaces hydroxyl groups in wood lignin with acetyl groups, and as a result, the material is more dimensionally stable, and the natural durability is improved (Rowell *et al.* 1993; 1994). Furfurylation is based upon the reaction of wood with the bio-chemical furfuryl alcohol at the cell wall level. Both processes significantly improve wood properties, protecting against decay, decreased hygroscopicity and improved dimensional stability (Jones and Hill 2007). Acetylation and furfurylation have not been implemented for bamboo material. Sugiyanto (2011) reported that both methods are potential for enhancing bamboo sheets properties.

Weathering performance is defined as the extent of degradation after the material is exposed to outdoor conditions. In an outdoor climate, the material is in direct contact with solar radiation, moisture, heat, atmospheric

pollutants and microorganisms and components are thus degrade with exposure (Pospisil and Nespurek 2000; Xie *et al.* 2005). Lignocellulosic materials such as wood and bamboo are responsive to outdoor environmental factors (Feist 1983). The use of bamboo in outdoor conditions has not been extensively studied (Liese 1987). Similar to wood, photo-degradation and moisture changes during exposure are the predominant factors driving bamboo degradation. During exposure, uneven shrinkage and swelling lead to the surface checking of bamboo (Feist 1983; Kim *et al.* 2008). Kim *et al.* (2008) reported that exposing bamboo culm to outdoor conditions for less than 12 months transformed its colour to grey with numerous checks occurring. Anatomically, most parenchyma tissues are degraded. Lignin rapidly decreases during the initial weathering period (Kim *et al.* 2008). The effect of weathering upon chemically modified bamboo has not been studied intensively.

Two essential parameters of weathering performance include colour changes and moisture sorption. Colour differences due to weathering can be measured quantitatively using a colourimetry based system characterized by three parameters: L^* , a^* and b^* for easy quantification. The L^* value represents the lightness, while a^* and b^* are the redness and yellowness values. The total colour changes (ΔE^*) due to weathering is precisely measured before and after weathering (Zhang *et al.* 2009). Moisture sorption in the wood after exposure can be determined based upon its surface wettability (Kalnins and Feist 1993). Surface wettability can be determined based upon the contact angle between the liquid and wood surfaces. Two techniques available for contact angle measurement include sessile drops and tilting plate methods. The sessile drop method involves placing a drop

of water on the wood surface and measuring the contact angle at a specific time (Wellons 1980; Kalnins and Feist 1993). The tilting plate method involves immersion of the flat wood surfaces in distilled water followed by tilting until the wood-water interface is no longer curved (Freeman 1959; Bodig 1962). A sessile drop is a simple way to measure contact angle and accordingly has been widely used to measure the contact angle of exposed wood (Kalnins and Feist 1993). This paper evaluates the weathering performance of furfurylated and acetylated bamboo sheets. Parameters studied include colour changes and contact angle after accelerated weathering process at QUV chamber.

Materials and Methods

Sample Preparation

Bamboo strips were prepared from three years old petung bamboo (*Dendrocalamus asper* (Schultes f.) Backer ex Heyne). To fit the QUV/Spray V-230 A8 chamber, bamboo sheets (75 x 4 x 3 mm) were cut from bamboo culm (Fig. 1). Bamboo samples were matched from the same internodes and grouped into three sets (acetylated, furfurylated and non-modified). Bamboo was acetylated and furfurylated based upon the treatment methods, as mentioned by Sugiyanto (2011). Only the optimum level of chemical modification was tested: 16% weight percentage gain for acetylated samples and 76% for furfurylated samples. After modification, all samples together with non-modified bamboo were placed in a conditioning room (25°C, 65% relative humidity) until the equilibrium moisture content was reached within three weeks.

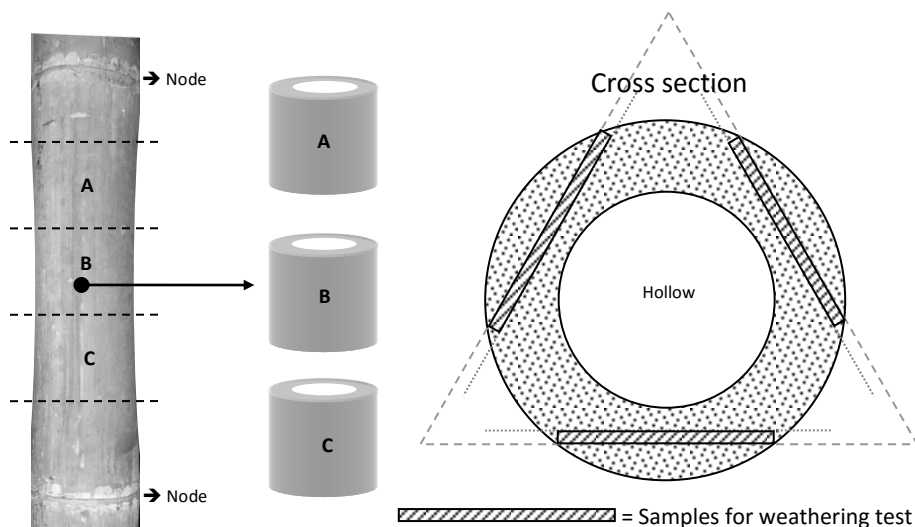


Figure 1. Sample pattern for weathering test.

Accelerated Weathering Test

The main weathering forces that cause material degradation include sunlight, high temperature and moisture exposure (Q-Lab 2006). Outdoor weathering tests are time-consuming, and such forces vary considerably producing significantly variable results. Consequently, various chambers designed to mimic accelerated weathering conditions have been developed to test material behaviour in outdoor use (Q-Lab 2006).

The QUV/Spray V-230 A8 accelerated weathering chamber has been developed for testing materials under specific outdoor conditions (Q-Lab 2006). The QUV/Spray V-230 A8 chamber can reproduce the damage that may occur over months or even years of outdoor exposure in hours. The apparatus is equipped with UVA-340 lamps to simulate shortwave sunlight within the chamber. The chamber also simulates exposure to moisture as the dew, with cycles of heating and condensation. The apparatus is equipped with water sprays to mimic the effects of rain as

well as thermal shock and erosion (Q-Lab 2006). Section 12 examines the impact of weathering upon acetylated and furfurylated bamboo sheets using the QUV/Spray V-230 A8 accelerated weathering chamber. Colour change and contact angle differences (by the sessile drop method) are investigated. The QUV/Spray V-230 A8 weathering chamber was used for weathering accelerated testing. Samples were subjected to accelerated weathering by exposure to 340 nm fluorescent UV lamps (Fig. 2). The weathering schedule involved continuous light irradiation (2 hours) followed by water spraying (18 minutes) (ASTM International 2010). The average irradiance was 0.85 W/m^2 at 340 nm wavelength with a chamber temperature of 45°C . The output of a 340 nm lamp is concentrated in the UV region ranging from 300–400 nm with an apex at 340 nm. Ninety-six bamboo sheets, mounted on the sample holder (Fig. 2) were monitored every 300 hours for a total of 1200 hours. Each inspection consisted of colour and contact angle measurements.



Figure 2. The QUV/Spray V-230 A8 weathering chamber and samples mounted on the aluminium sample holder of the chamber slots.

Colour Measurement

Colour values before and after weathering were measured using a Minolta CR-300 tristimulus colour measuring instrument. The colour value was presented using the *Commission International de l' Eclairage* (CIE) 1976 (L^* , a^* , b^*) colour space, also known as the CIELAB system. There are three coordinates: L^* represents

lightness (100 = white, 0 = black), a^* value represents redness ($+a^*$ = red, $-a^*$ = green) and b^* represents yellowness ($+b^*$ = yellow, $-b^*$ = blue). Total colour changes (ΔE^*) value were calculated based on L^* , a^* and b^* value differences (Equation 1 and 2). A low ΔE^* corresponds to a low colour change or a stable colour.

$$\Delta L^* = L^*_f - L^*_i; \Delta a^* = a^*_f - a^*_i;$$

$$\Delta b^* = b^*_f - b^*_i \dots\dots\dots(1)$$

$$\Delta E^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \dots\dots\dots(2)$$

Where ΔL^* , Δa^* and Δb^* are the changes between the initial (*i*) and the final (*f*) values. ΔE^* is the total colour changes.

Contact Angle Measurement

Contact angles before and after weathering exposure were measured using the sessile drop technique. Twenty-five-micron litre (μ l) of distilled water was dropped onto the bamboo surface and recorded using a Dyno 3111 S digital video camera (Fig. 3). Measurement of contact angles was made on still video images, and quantification of the angle was recorded based upon the digital images using 'Image-J' software (Rasband 2003). For consistency, contact angle comparisons were made 10 seconds after dropping time.

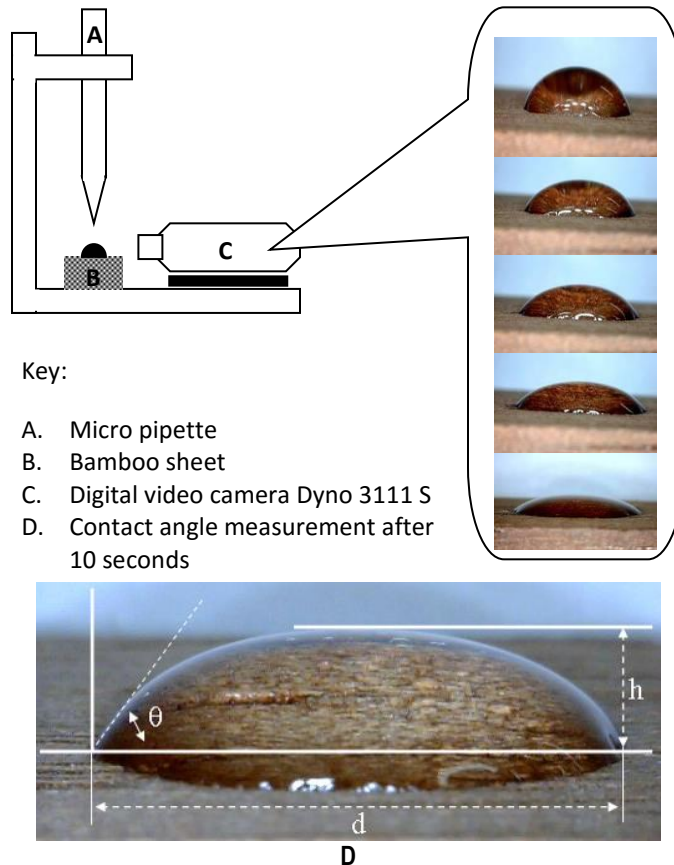


Figure 3. Contact angle measurement using the sessile drop technique.

Results and Discussion

Colour Differences

The average colour value differences of chemically modified and non-modified bamboo sheets after weathering are presented in Table 1.

Table 1 shows that after weathering, the greatest ΔE^* was recorded in furfurylated sheets, while ΔE^* of acetylated sheets achieved the lowest value. Less colour value differences (ΔE^*) indicates less change of colour

due to weathering. That is, less ΔE^* means colour stability of bamboo sheets after exposure. The colour value differences between bamboo sheets increased with longer exposure time. The greatest lightness differences ΔL^* was recorded in furfurylated bamboo sheets, while the ΔL^* of acetylated and non-modified sheets were similar. The increase of ΔL^* (lightness value) indicates most sheets tend to be whitish after exposure. The redness (a^*) and yellowness (b^*) values of weathered sheets were negative, meaning the sheets turned greenish and bluish after

exposure. In combination between L^* , a^* and b^* values, the sheets turn to grey after weathering.

The normality of data set (Shapiro-Wilk) (Table 2) shows that the colour value differences before and after weathering of bamboo strips conformed with a normal distribution except the weathering of non-modified strips for 600 hours. Therefore, a one-way ANOVA was applied in the case of normally distributed data, while Kruskal-Wallis one-way ANOVA with ranks were examined for non-Gaussian data (Field 2005).

The results of a two-way ANOVA performed on groups of chemically modified strips (acetylation, furfurylation and non-modified) and weathering time (300, 600, 900 and 1200 hours) into the total colour differences (ΔE^*) demonstrated significant differences between treatments ($F = 41.116$, $p < 0.001$), while there were no significant differences among weathering exposure time ($F = 2.661$, $p = 0.053$). The total colour differences (ΔE^*) of bamboo strips after weathering is shown in Fig. 4.

Table 1. The average colour value differences after weathering.

Sample groups	Weathering exposure time (hours)							
	300		600		900		1200	
	<i>x</i>	<i>sd</i>	<i>x</i>	<i>sd</i>	<i>x</i>	<i>sd</i>	<i>x</i>	<i>sd</i>
$\Delta L^* (unit)$								
Non-modified	3.9	0.6	2.4	0.7	3.2	1.3	2.8	1.5
Acetylated	3.8	0.8	2.3	1.3	3.1	0.9	3.2	1.1
Furfurylated	5.4	0.3	5.6	0.5	5.8	1.1	5.7	0.4
$\Delta a^* (unit)$								
Non-modified	-1.2	0.1	-1.3	0.1	-1.5	0.2	-1.5	0.1
Acetylated	-0.7	0.1	-0.9	0.5	-1.3	0.5	-1.4	0.3
Furfurylated	-0.8	0.1	-0.6	0.3	-0.7	0.2	-0.8	0.4
$\Delta b^* (unit)$								
Non-modified	-3.4	0.3	-4.3	1.3	-3.9	1.3	-4.2	1.2
Acetylated	-1.6	0.2	-3.7	1.1	-3.3	1.0	-2.5	1.4
Furfurylated	-1.4	0.2	-2.2	0.6	-2.1	0.8	-2.2	0.7
$\Delta L^* (unit)$								
Non-modified	5.3	0.5	5.3	0.9	5.5	0.3	5.6	0.3
Acetylated	4.2	0.7	4.6	1.1	4.8	1.0	4.6	0.4
Furfurylated	5.6	0.3	6.1	0.3	6.3	0.9	6.3	0.3

Remarks: *x*=mean; *sd*=standard deviation

Table 2. Results of Shapiro-Wilk test for colour changes (ΔE^*) of bamboo strips after weathering.

Colour differences	Accelerated weathering test (hours)			
	300	600	900	1200
<i>Non-modified</i>				
ΔL	0.890 *	0.766	0.990 *	0.950 *
Δa	0.944 *	0.788	0.833 *	0.979 *
Δb	0.936 *	0.773	0.919 *	0.879 *
ΔE	0.890 *	0.766	0.990 *	0.950 *
<i>Acetylated</i>				
ΔL	0.782	0.945 *	0.899 *	0.782
Δa	0.908 *	0.832 *	0.945 *	0.884 *
Δb	0.934 *	0.950 *	0.969 *	0.979 *
ΔE	0.782	0.945 *	0.899 *	0.782
<i>Furfurylated</i>				
ΔL	0.968 *	0.955 *	0.878 *	0.911 *
Δa	0.917 *	0.875 *	0.926 *	0.872 *
Δb	0.943 *	0.989 *	0.957 *	0.882 *
ΔE	0.968 *	0.955 *	0.878 *	0.911 *

Remarks: * indicates data matches with the pattern of normal distribution

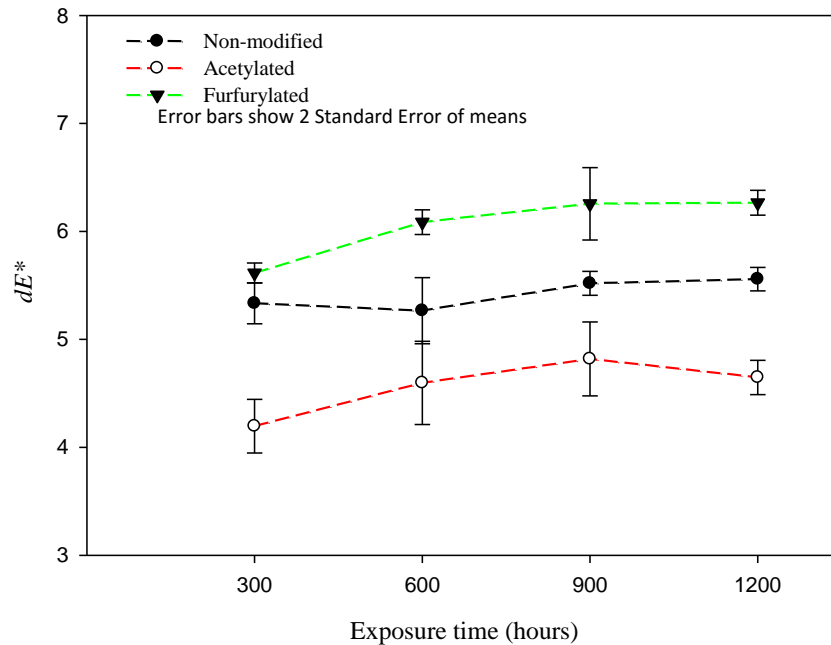


Figure 4. Average values of total colour differences (ΔE^*) of bamboo strips after accelerated weathering.

Fig. 4 shows that the total colour differences (ΔE^*) of furfurylation are greater than non-modified strips, while colour differences of acetylated bamboo strips is less than non-modified strips. The difference in the mean values of treated bamboo strips is greater than would be expected by chance; as a result, there is the statistical difference ($p < 0.001$). The difference in mean values of exposure time is not significant enough to exclude the possibility that the difference is just due to random sampling variability. The graph in Fig. 4 also shows the colour difference tends to increase with longer exposure time.

Similar to wood, bamboo naturally contains several chromophoric functional groups and an aromatic skeleton, making it a good absorber of light. During exposure to solar radiation, which includes ultraviolet light, various reactions are initiated with lignocellulosic components (primary and secondary hydroxyl, carbonyl, carboxyl, aromatic and phenolic groups) (Evans *et al.* 2005; Xie *et al.* 2005). Photochemical degradation is evident by changes in colour to yellow or brown, which proceed to an eventual greying (Chang *et al.* 1982; Feist 1983).

In all bamboo sheets examined, colour differences ΔE^* values are higher than 1.5, which means colour differences can be visually distinguished by the human eye (Horváth and Halász-Fekete 2005). After weathering, bamboo strip colour tended to become greyish. Similarly, with that of weathered wood, the greyish colour after exposure confirmed the photo-degradation of lignin, extractives and bonded chemical (Zhang *et al.* 2009). Slowing down of lignin photo-degradation by acetylation is

attributed to the acetyl groups which limit the lignin degradation.

Contact Angle (Wettability)

The average contact angle values are shown in Table 3.

Table 3. Average contact angle values.

Bamboo sheets	Exposure (hours)	Contact angle (°)	
		\bar{x}	sd
Non-modified	0	54.0	0.9
	300	35.0	1.3
	600	31.1	0.8
	900	30.8	0.6
	1200	30.1	0.8
Furfurylated	0	53.6	1.3
	300	47.3	0.7
	600	43.3	0.7
	900	42.2	1.6
	1200	42.3	1.3
Acetylated	0	53.5	1.3
	300	51.3	0.8
	600	51.7	1.4
	900	50.6	1.0
	1200	50.7	1.2

Remarks: \bar{x} =mean; sd =standard deviation.

Table 3 shows the average contact angle value decreased with longer exposure time. Weathered samples

exhibited lower contact angles than the initial samples, indicating that bamboo sheets easily absorb water after weathering. The average contact angle of non-modified bamboo sheets decreased significantly from 54° to 30° after 1200 hours of exposure. The average contact angle of furfurylated sheets decreased slightly from 54° to 42° after 1200 hours of exposure. The contact angle in acetylated bamboo sheets reduced from 53° to 50° after 1200 hours of exposure.

The measured contact angle at 10 seconds after dropping time on the weathered sheet's surfaces, as a

function of the exposure duration, is presented in Fig. 5. Normality test (Shapiro-Wilk) of contact angles showed that the data were normally distributed; therefore, a two-way ANOVA was performed. The results of two-way ANOVA between modified strips (acetylated, furfurylated and non-modified) and exposure time (300, 600, 900 and 1200 hours) showed that both factors (treatment and exposure time) were significantly different with $F = 2501.3$ ($p < 0.001$) for treatment and $F = 704.41$ ($p < 0.001$) for exposure time.

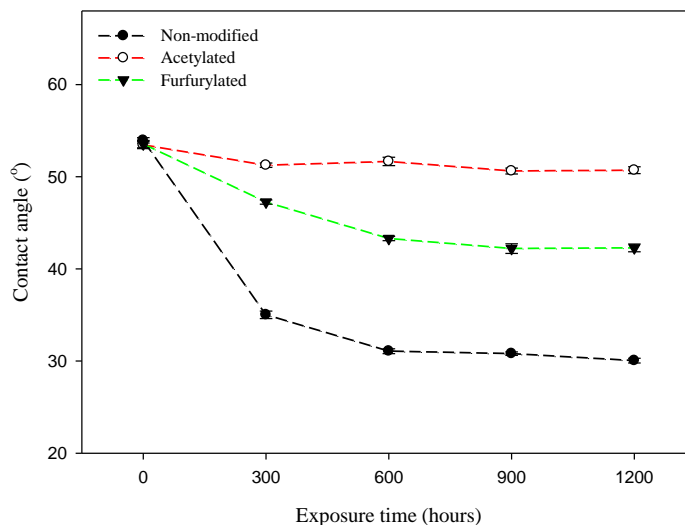


Figure 5. The contact angle of non-modified and chemically modified bamboo sheets after accelerated weathering

Fig. 5 shows the contact angle measurement of bamboo sheets after accelerated weathering. Weathered, non-modified sheets exhibited lower contact angles than chemically modified sheets. Weathering, through the action of artificial sunlight and water spray in the QUV/Spray V-230 A8 chamber, caused gradual destruction of the bamboo sheets. Most bamboo tissues were degraded, and non-modified sheets easily absorbed water following weathering. These findings are in accord with those of Kim *et al.* (2008) who found most ground parenchyma was degraded during initial weathering and the bamboo surface became more wettable, and water repellency was reduced.

The initial contact angle of non-modified bamboo sheets was around 54° and decreased to 35° after 300 hours of exposure. After a further 300 hours of exposure, the contact angle was further reduced to 30°. Contact angles did not significantly change in subsequent examinations (900 and 1200 hours). In wood, Kalnins and Feist (1993) attributed decreases in contact angle to the photo-oxidation of extractives to volatile products and the removal of water-soluble compounds. The value of the contact angle of chemically modified sheets confirmed that acetyl groups and furan polymer slow down the wettability of the weathered bamboo sheets surfaces.

Lignin and extractives are known to be partially responsible for the water repellency of wood and the high hydrophilic property of cellulose (Rowell and Banks 1985). Any slight modification of lignin and extractives in the wood will contribute to the alteration of the contact angle. The reduction in the contact angle of weathered bamboo sheets is a strong indication that weathering promotes the degradation of lignin and extractives.

The contact angles of both acetylated and furfurylated bamboo sheets were also reduced but not to the same magnitude as non-modified sheets. The angles of acetylated sheets were decreased by 2–4° after 300 and 600 hours of exposure but were not significantly reduced after 900 and 1200 hours of exposure. The contact angle of furfurylated bamboo sheets rapidly fell by 6° in the first 300 hours but diminished after 600, 900 and 1200 hours of exposure. The contact angles of chemically modified bamboo sheets were higher than non-modified bamboo sheets after weathering. That is, chemically modified bamboo sheets did not easily absorb water after weathering.

Similar to those of wood, during weathering bamboo sheets exposed to rapid moisture changes during outdoor exposure (Xie *et al.* 2005). Water from rain or dew on the wood surface is quickly absorbed by capillarity on the surface layer, followed by adsorption within wood cell walls.

Water vapour is taken up directly by the wood by adsorption under increased relative humidity and causes the swelling. Uneven swelling between the surface and interior may result in surface warping, cupping and surface checking after outdoor exposure (Feist 1983).

In the case of wood, the exposure to such areas allows the initiation of degradation during outdoor exposure. To protect the wood in outdoor conditions, treatment with acetic anhydride has been used. Plackett *et al.* (1992) reported that acetylated wood was adequately protected from degradation due to decreased moisture sorption. Wood discolouration due to weathering cannot, however, be reduced (Plackett *et al.* 1992).

Conclusions

The study shows that the total colour differences (ΔE^*) of furfurylation are greater than non-modified strips, while colour differences of acetylated bamboo strips are less than non-modified strips. To summarize, chemically modified sheets turn grey after weathering. Slowing of lignin photo-degradation by acetylation is attributed to the acetyl groups, which limits the degradation of lignin. Treating bamboo sheets with acetic anhydride and furfuryl alcohol was found to be effective in protecting bamboo from absorbing water during weather exposure.

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WOOD RESEARCH Journal

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4. Other rules:
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 - 4.2. Values between are written using this symbol ($\frac{\square}{\square}$), e.g. 3.75 $\frac{\square}{\square}$ 8.92%.
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Example of Table and Figure

Table 1. Effects of temperature on *in vitro* growth of seedlings.

Temp. (°C)	Shoot length (mm)	Number of leaf	Fresh weight (g)
25	59.2 ± 10.6 ^c	4.5 ± 0.8 ^a	0.29 ± 0.13 ^a
27	88.5 ± 9.3 ^a	4.8 ± 0.9 ^a	0.40 ± 0.12 ^a
29	75.0 ± 11.1 ^b	3.8 ± 0.6 ^a	0.30 ± 0.07 ^a

Note: Values (average ± standard deviation) with different letters are statistically significant according to Tukey's multiple comparison test. Data were recorded after 4 weeks of culture. MS medium was used as a basal medium without any PGRs. Number of sample = 10.

Source: Chujo *et al* 2010.

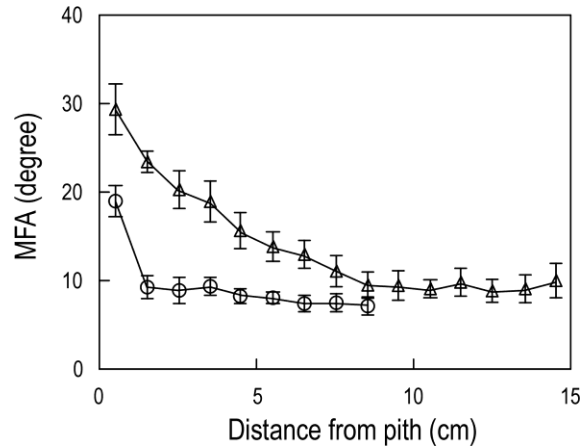


Figure 3. Radial variation of microfibril angle of the S2 layer in tracheid. Open circle, *Agathis* sp.; open triangle, *Pinus insularis*; Bars indicate the standard deviation. (Source: Ishiguri *et al* 2010)

