

WOOD RESEARCH Journal

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Application of Single and Double Component Wood Bleaching Chemicals on Surface Properties of Naga (*Brachystegia cynometroides*) Wood

Hüseyin Peker, Göksel ULAY, & Ümit Ayata

Abstract

In this study, some surface changes (color parameters, whiteness index (W^*), and glossiness) occurring after the application of single [oxalic acid ($C_2H_2O_4$)] and double component [sodium hydroxide (NaOH) + hydrogen peroxide (H_2O_2)] wood bleaching chemicals on naga (*Brachystegia cynometroides*) wood were investigated. The bleached surfaces were compared with the unbleached surfaces. According to the obtained results, the ΔE^* values were determined as 2.99 for $C_2H_2O_4$ chemical and 16.95 for NaOH + H_2O_2 chemical. Significant results were found for all tests in the analysis of variance. Increases in b^* and C^* values were observed with $C_2H_2O_4$, while decreases were observed with NaOH + H_2O_2 chemical. With both wood bleaching chemicals, increases were found in W^* values in parallel directions to the fibers, as well as in h^o and L^* values, while decreases were obtained in glossiness values at 60 and 85 degrees in both parallel and perpendicular directions to the fibers, and in a^* values.

Keywords: Naga, bleaching, whiteness index, *Brachystegia cynometroides*, color, glossiness

Introduction

The color of wood is an important indicator in the assessment of surface characteristics and the value of items. Therefore, wood bleaching has become one of the advanced technologies for enhancing the visual features and decorative effects of wood, as well as for the effective use of man-made rapidly growing wood (Duan 2002).

Bleaching is a process of color removal or whitening that can occur in a solution or on a surface. Materials that produce color in the solution or on fibers typically consist of organic compounds with broad conjugated chains containing alternating single and double bonds and typically including heteroatoms, carbonyl, and phenyl rings in the conjugated system. The part of the molecule that absorbs a light photon is called a chromophore (color carrier). Bleaching agents are used not only in textile, paper, and pulp bleaching but also in home laundry (Farr *et al.* 2003).

There are two methods for bleaching wood. The first method involves commercial wood bleaches that are high-strength acidic bleaches. These come with various brand names and can be purchased from most paint and hardware stores. The second bleaching method is household chlorine bleach. Bleaching wood takes longer but is much safer to use and causes less damage to the wood (Broadnax 1977).

A moderate increase in brightness or decrease in color can be achieved in a single step. The higher the target color removal, the more stages are required, increasing the likelihood of damaging the product's properties. This is particularly true for delicate materials (Suess 2009).

Under mild operating conditions, hydrogen peroxide has little or no lime-removing effect, making it particularly attractive for bleaching highly efficient (mechanical and semi-chemical) pulps. However, under severe conditions, this

reagent has delignifying properties that can be used to obtain chemically pulped paper with high brightness and stability with very little degradation. Hydrogen peroxide is easy to transport, store, and use. It is highly flexible and almost non-volatile. Additionally, its reaction products are relatively non-toxic (Ramos *et al.* 2008).

Bleaching is the most important step in the experiment. At this stage, the linkage of conjugated double bonds in the side chain of lignin has been broken, as well as the chromophoric groups, resulting in a whiter absorption spectrum moving from the visible region to the ultraviolet region. It is advantageous to put it into an alkaline solution at this stage, but in a strong alkaline solution, H_2O_2 decomposes and releases O_2 , which increases the use of H_2O_2 . If the pH drops below 9, the decomposition of H_2O_2 slows down significantly. It is very important to control the pH in a narrow range (Hemmasi 2017).

In the literature, there are studies on bleaching applications using different bleaching chemicals on various wood species (Ayata and Bal 2024; Ayata and Çamlıbel, 2024; Ayata *et al.* 2024a,b; Çamlıbel and Ayata 2024; Peker *et al.* 2023a,b,c; 2024; Yamamoto *et al.* 2017; Ayata and Çamlıbel 2023; 2024; Mononen *et al.* 2005; Ayata and Bal 2023; Nguyen *et al.* 2019; Peker and Ayata 2023; Mehats *et al.* 2021; Peker 2023a,b; Peker and Ulusoy 2023; Ayata 2024; Liu *et al.* 2015; Çamlıbel and Ayata 2023a,b). However, it has been observed that no bleaching application has been performed on naga wood.

Naga can sometimes reach heights of up to 40 meters, with a cylindrical trunk (Aubréville, 1936). The heartwood is light to dark brown, somewhat resembling low-quality mahogany. It may have alternating dark and light streaks. The sapwood is about 15 cm wide, white or pale in color, and distinct from the heartwood. The fibers are generally tightly

interlocked, and quarter-sawn surfaces display a ribbon-like figure. The texture is medium (Anonymous, 1956).

Cutting this timber with machine tools is moderately difficult, while working with hand tools is challenging. Saw teeth dull quite quickly. Most other processes result in moderate blunting of cutting edges. Due to the interlocked grain, severe tearing can occur during planing and molding. Achieving smooth surfaces is difficult, making the wood suitable for surface treatments. There is a moderate tendency for splitting during nailing. The wood is not suitable for tool manufacturing or general construction requiring high durability, but it is technically suitable for plywood production (Anonymous, 1956; 1951).

In this study, some surface changes occurring after the application of single and dual-component wood bleaching chemicals on naga (*Brachystegia cynometroides*) wood were investigated.

Materials and Methods

Wood Material

Samples of Naga (*Brachystegia cynometroides*) wood were prepared in dimensions of 100 mm x 100 mm x 15 mm. Conditioning treatments were applied to the samples (20±2°C with 65% relative humidity) (ISO 554, 1976).

Wood Bleaching Chemicals

Single-component (oxalic acid: C₂H₂O₄) and dual-component (hydrogen peroxide (H₂O₂) and sodium hydroxide (NaOH), prepared in a 2:1 ratio) bleaching chemicals were used.

Application of Bleaching on Wood Surfaces

Before the bleaching application, the wooden material surfaces were sanded with 80, 100 and 150 grit sandpaper. These chemicals were applied to the wood surfaces using a sponge, in a single layer. After applying one coat, the treated wood was allowed to dry. Then measurements were taken.

Determination of Some Surface Properties

Whiteness index (*W*^{*}) values (ASTM E313-15e1 2015) (Whiteness Meter BDY-1), glossiness tests (ISO 2813 1994) (ETB-0833 model gloss meter), shore D hardness values (ASTM D 2240 2010), and color changes (ASTM D 2244-3 2007) (CS-10) were measured. Total color differences were calculated using the following formulas.

$$\Delta L^* = (L^*_{\text{bleached sample}} - L^*_{\text{unbleached sample}}) \quad (1)$$

$$C^* = [(a^*)^2 + (b^*)^2]^{0.5} \quad (2)$$

$$h^\circ = \arctan(b^*/a^*) \quad (3)$$

$$\Delta H^* = [(\Delta E^*)^2 - (\Delta L^*)^2 - (\Delta C^*)^2]^{0.5} \quad (4)$$

$$\Delta C^* = (C^*_{\text{bleached sample}} - C^*_{\text{unbleached sample}}) \quad (5)$$

$$\Delta a^* = (a^*_{\text{bleached sample}} - a^*_{\text{unbleached sample}}) \quad (6)$$

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{0.5} \quad (7)$$

$$\Delta b^* = (b^*_{\text{bleached sample}} - b^*_{\text{unbleached sample}}) \quad (8)$$

ΔC^* : chroma difference or saturation difference, and ΔH^* : hue difference or shade difference are indicated. Table 1 provides important information on other parameters (Lange, 1999). ΔE^* comparison criteria (DIN 5033 1979) are given in Table 2.

Table 1. Information regarding Δa^* , ΔL^* , Δb^* , and ΔC^* values (Lange 1999)

Test	Positive Description	Negative Description
Δb^*	More yellow than the reference	More blue than the reference
ΔL^*	Lighter than the reference	Darker than the reference
Δa^*	More red than the reference	More green than the reference
ΔC^*	Clearer, brighter than the reference	More dull, matte than the reference

Table 2. Comparison criteria for ΔE^* assessment (DIN 5033 1979)

Visual	Total Colour Difference	Visual	Total Colour Difference
Undetectable	<0.20	Distinct	1.50 - 3.00
Very Weak	0.20 - 0.50	Very Distinct	3.00 - 6.00
Weak	0.50 - 1.50	Strong	6.00 - 12.00
		Very Strong	> 12.00

Statistical Analysis

Standard deviations, means, minimum and maximum values, homogeneity groups, variance analysis results, and percentages (%) of changes have been calculated using a statistical program.

Table 3. Results of shore D hardness values

Number of Measurements	Mean (HD)	Standard Deviation	Minimum	Maximum	Coefficient of Variation
10	49.30	2.58	47.00	54.00	5.24

The ΔE^* values were calculated as 2.99 with $C_2H_2O_4$ chemical and 16.95 with $NaOH + H_2O_2$ chemical. For both bleaching agents, the Δa^* values were negative (greener than the reference) and the ΔL^* values were positive (lighter than the reference) as determined (Table 4).

With $C_2H_2O_4$ chemical, the ΔC^* and Δb^* values were positive (clearer, brighter, and more yellow than the

Results and Discussion

The Shore D test result for wood is obtained as 49.30 HD (Table 3). The results of ΔE^* are presented in Table 4.

reference), while with $NaOH + H_2O_2$ chemical, they were negative (duller, cloudier, and more blue than the reference) (Table 4).

"Distinct (1.5 to 3.0)" criteria were obtained with $C_2H_2O_4$ chemical and "very strong (> 12.0)" with $NaOH + H_2O_2$ chemical (Table 4).

Table 4. Results of total color differences

Chemical Type	ΔL^*	Δa^*	Δb^*	ΔC^*	ΔH^*	ΔE^*	The color change criteria (DIN 5033, 1979)
$C_2H_2O_4$	0.16	-0.11	2.98	2.40	1.77	2.99	Distinct (1.5 ila 3.0)
$NaOH + H_2O_2$	13.07	-9.89	-4.32	-8.55	6.58	16.95	Very Strong (> 12.0)

The test results are provided in Table 5. With the application of bleaching agents to wooden material surfaces, increases were observed in the direction parallel to the fibers for both bleaching agents in terms of W^* , h^* , and L^* values, while decreases were noted in the glossiness values and a^* values in both perpendicular and parallel directions at 60 and 85 degrees. Additionally, increases were observed with single component in b^* and C^* values, while decreases were observed with $NaOH + H_2O_2$ chemical.

The highest results in brightness values and a^* values in both perpendicular and parallel directions at 60 and 85 degrees were found in the control experiment group. For W^* , increases were obtained with $NaOH + H_2O_2$ chemical in glossiness values perpendicular to the fibers and at 20 degrees perpendicular and parallel to the fibers, while decreases were obtained with $C_2H_2O_4$ chemical (Table 5).

The lowest results for L^* (62.20), h^* (52.54), and W^* (10.65) values were observed in the control test samples. The lowest results for a^* (6.20), b^* (16.69), and C^* (17.87) values were obtained with $NaOH + H_2O_2$, whereas the lowest W^* \perp value (20.04) was determined with $C_2H_2O_4$ chemical (Table 5).

When considering the highest results, L^* (75.27), h^* (69.66), and W^* (\perp : 37.32 and \parallel : 24.78) values were obtained with $NaOH + H_2O_2$ chemical, while the a^* (16.09) test was found with the control samples, and the b^* (23.98) and C^* (28.82) tests were found with $C_2H_2O_4$ chemical (Table 5).

Table 5. Changes in color parameters, glossiness values, and whiteness index values after the application of single and dual-component wood bleaching chemicals on naga (*Brachystegia cynometroides*)

Test	Chemical Type	Mean	Change (%)	HG	Standard Deviation	Minimum	Maximum	COV
L^*	Control	62.20	-	B**	0.31	61.72	62.57	0.50
	$C_2H_2O_4$	62.35	\uparrow 0.24	B	0.35	61.71	62.85	0.57
	$NaOH + H_2O_2$	75.27	\uparrow 21.01	A*	1.06	73.80	76.76	1.40
a^*	Control	16.09	-	A*	0.20	15.72	16.45	1.22
	$C_2H_2O_4$	15.98	\downarrow 0.68	A	0.37	15.48	16.66	2.29
	$NaOH + H_2O_2$	6.20	\downarrow 61.47	B**	0.68	5.56	7.18	11.02
b^*	Control	21.00	-	B	0.24	20.68	21.33	1.13
	$C_2H_2O_4$	23.98	\uparrow 14.19	A*	0.28	23.46	24.49	1.17
	$NaOH + H_2O_2$	16.69	\downarrow 20.52	C**	0.99	15.70	18.13	5.96

C*	Control	26.42	-	B	0.27	25.99	26.80	1.01
	C ₂ H ₂ O ₄	28.82	↑9.08	A*	0.39	28.11	29.27	1.37
	NaOH + H ₂ O ₂	17.87	↓32.36	C**	1.13	16.74	19.50	6.32
h ^o	Control	52.54	-	C**	0.39	51.93	53.03	0.75
	C ₂ H ₂ O ₄	56.32	↑7.19	B	0.48	55.27	56.80	0.85
	NaOH + H ₂ O ₂	69.66	↑32.58	A*	1.05	68.28	71.45	1.51
⊥20°	Control	0.50	-	B	0.00	0.50	0.50	0.00
	C ₂ H ₂ O ₄	0.48	↓4.00	B**	0.04	0.40	0.50	8.78
	NaOH + H ₂ O ₂	0.66	↑32.00	A*	0.14	0.40	0.80	21.66
⊥60°	Control	2.66	-	A*	0.20	2.50	3.00	7.35
	C ₂ H ₂ O ₄	1.96	↓26.32	C**	0.08	1.90	2.10	4.30
	NaOH + H ₂ O ₂	2.22	↓16.54	B	0.08	2.10	2.30	3.55
⊥85°	Control	1.10	-	A*	0.30	0.80	1.60	27.10
	C ₂ H ₂ O ₄	0.10	↓90.91	B**	0.00	0.10	0.10	0.00
	NaOH + H ₂ O ₂	0.10	↓90.91	B**	0.00	0.10	0.10	0.00
20°	Control	0.50	-	B	0.00	0.50	0.50	0.00
	C ₂ H ₂ O ₄	0.44	↓12.00	B**	0.11	0.30	0.60	24.43
	NaOH + H ₂ O ₂	0.64	↑28.00	A*	0.05	0.60	0.70	8.07
60°	Control	3.38	-	A*	0.18	3.10	3.60	5.37
	C ₂ H ₂ O ₄	2.20	↓34.91	C**	0.09	2.10	2.30	4.29
	NaOH + H ₂ O ₂	2.70	↓20.12	B	0.13	2.60	2.90	4.94
85°	Control	2.72	-	A*	0.28	2.50	3.20	10.22
	C ₂ H ₂ O ₄	0.58	↓78.68	B	0.21	0.30	0.80	37.07
	NaOH + H ₂ O ₂	0.10	↓96.32	C**	0.00	0.10	0.10	0.00
W [*] (⊥)	Control	21.55	-	B	0.26	21.20	21.90	1.22
	C ₂ H ₂ O ₄	20.04	↓7.01	C**	0.17	19.80	20.30	0.85
	NaOH + H ₂ O ₂	37.32	↑73.18	A*	1.68	35.10	39.20	4.50
W [*] ()	Control	10.65	-	B**	0.14	10.50	10.80	1.27
	C ₂ H ₂ O ₄	10.86	↑1.97	B	0.14	10.70	11.00	1.32
	NaOH + H ₂ O ₂	24.78	↑132.68	A*	0.96	23.70	26.00	3.87

HG: Homogeneity Group, COV: Coefficient of Variation, Number of Measurements: 10, *: Highest value, **: Lowest value

Variance analysis results are provided in Table 6. Upon examination of the variance analysis results, the type of bleaching agent was found to be significant for all tests (Table 6).

Table 6. Analysis of variance results (*: Significant)

Test	Sum of Squares	df	Mean Square	F value	Sig.
L*	1125.402	2	562.701	1261.386	0.000*
a*	644.971	2	322.485	1514.875	0.000*
b*	269.124	2	134.562	358.953	0.000*
C*	663.118	2	331.559	662.482	0.000*
h ^o	1618.038	2	809.019	1619.687	0.000*
⊥20° glossiness	0.195	2	0.097	13.140	0.000*
⊥60° glossiness	2.504	2	1.252	72.853	0.000*
⊥85° glossiness	6.667	2	3.333	112.500	0.000*
20° glossiness	0.211	2	0.105	22.219	0.000*
60° glossiness	7.016	2	3.508	176.709	0.000*
85° glossiness	38.915	2	19.457	472.435	0.000*
W [*] (⊥)	1831.905	2	915.952	941.584	0.000*
W [*] ()	1311.558	2	655.779	2048.124	0.000*

Various chemicals used in bleaching studies have been reported to induce changes in color parameters (Peker *et al.* 2023a;b;c; 2024; Yamamoto *et al.* 2017; Ayata and Çamlıbel 2023; 2024; Mononen *et al.* 2005; Ayata and Bal 2023; Nguyen *et al.* 2019; Peker and Ayata 2023; Mehats *et al.* 2021; Peker 2023a;b; Peker and Ulusoy 2023; Liu *et al.* 2015; Çamlıbel and Ayata 2023a;b).

Conclusions

The application of bleaching agents has resulted in changes in color parameters, glossiness values, and whiteness index values. These results have been confirmed by the data obtained from SPSS. The NaOH + H₂O₂ chemical had a greater effect on wood surfaces in terms of total color

difference results compared to the C₂H₂O₄ chemical. The obtained results are important in terms of the potential applications of this tree species. It is recommended to conduct various aging tests on these materials.

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Comparative Study of Flexural Behavior of Bolted-Laminated and Glue-Laminated Sengon Timber Beams (*Albizia falcataria*)

Yosafat Aji Pranata, Anang Kristianto, & Novi

Abstract

This study aimed to study the flexural behavior of bolt-laminated and glue-laminated sengon (*Albizia falcataria*) timber beams, which are flexural strength, modulus of rupture (MoR), and beam ductility. The study was conducted using an experimental testing method in the laboratory with reference to the ASTM D198. The number of test objects were three consecutively for bolt-laminated beams and glue-laminated beams. Results obtained from this study indicated that the flexural strength of bolt-laminated and glue-laminated sengon timber beams was not significantly different; the flexural strength of bolt-laminated beams were 3.1% higher compared to glue-laminated beams. The MoR and ductility of bolt-laminated beams were 8.4% and 14.2% higher compared to glue-laminated beams. These results indicate that the glue has an impact to the brittle behavior or limited ductility, while the bolts have an impact to make a more ductile beams. The general conclusion is that mechanical laminated timber technology can be an alternative to producing beams with larger cross-sectional sizes compared to solid timber, especially for low-grade wood, so that it can be used as part of the structural elements of buildings.

Keywords: Flexural strength, beam ductility, laminated timber, sengon, ASTM D198.

Introduction

Beams, as one of the main elements of a building besides columns, functioning for withstanding loads, both gravity and lateral loads, in the form of internal forces, namely bending moments and shear forces. The beam deflection must be designed not to exceed the permitted limit so that it meets the stiffness requirements. For the case of wide span beams, a larger cross-section is required according to the needs, while on the other hand, timber, especially low grades, is difficult to obtain because trees with large diameters are limited in number.

Timber lamination technology for building components, especially for beam elements, provides several benefits. It can be solutions for making beams with dimensions and cross-sectional sizes according to demands, thus it can be used as a structural component, for example, beams and columns for building construction. Examples of lamination technology are mechanical laminated timber with bolt that function as shear restraints and chemical laminated wood with glue as an adhesive between timber laminae.

A mechanical or bolt-laminated beam system is a beam composed of two or more laminae with bolts used as a shear-resistant connecting device. By having bolts installed at a certain distance, sliding that occurs between the laminae can be prevented (Pranata *et al.*, 2011). A glue lamination beam system is a beam composed of two or more laminae bonded with glue. The function of the glue is to prevent sliding between the laminae due to the load acting on the beam.

This study aims to study the flexural behavior of bolt-laminated and glue-laminated sengon (*Albizia falcataria*) timber beams, which are flexural strength, modulus of

rupture, and ductility ratio. Sengon trees are found in many tropical climates such as India, Vietnam, Thailand, Cambodia, Burma, Laos, China, and Indonesia. Sengon wood is classified as Strength Class III, with limited mechanical properties. Sengon trees are profitable in terms of production at a relatively shorter harvest period, which is around 3-6 years (Larasati, 2019; Prijono and Saputra, 2024), and as consequence, the diameter of log is also relatively small.

Researches have tried to increase the strength of sengon wood, namely by processing raw log materials into engineered wood product, for example, Laminated Veneer Lumber (LVL). Study on LVL high beams and its flexural capacity has been conducted (Putri and As'ad, 2015; Handayani, 2016; Awaludin *et al.*, 2018; Awaludin and Wusqo, 2021). Effendi and Awaludin (2022) studied the slender LVL beams, and research on LVL beams with non-prismatic cross-sections has also been conducted by Awaludin *et al.* (2019).

Further studies to form beams with a larger cross-sectional size according to needs by using glue lamination technology (chemical lamination) and bolt lamination (mechanical lamination) have been carried out. This product is used as a structural component for wide-span buildings. Beams are joined mechanically using fasteners (Fraserwood, 2024). Research on the flexural capacity, compressive capacity, and elastic modulus of sengon glue laminated wood has been conducted by Fakhri (2001), Lilis (2010), Wulandari and Ami (2022), Mutiara (2022), and Hadi and Lestari (2022). Table 1 shows the results of the flexural strength and the modulus of rupture of solid and glue laminated of sengon timbers.

Table 1. Flexural strength of sengon (*Albizia chinensis*) timber

Timber Products	References	F_b (MPa)	MoR (MPa)
Sengon (solid)	Wicaksono <i>et.al.</i> , 2017	-	20.65
Glue-Laminated Sengon	Anshari <i>et.al.</i> , 2018	-	24.31
Glue-Laminated Sengon	Chauf, 2019	14.00	17.00

Materials and Methods

The timber studied is Sengon (*Albizia falcataria*). The behaviors studied include flexural strength, modulus of rupture (MoR), and beam ductility. Flexural strength and MoR are parameters required for design of beam strength capacity, while ductility is a parameter required for design of beam stiffness.

There are several experimental testing methods for beam flexural testing. In this study, the four point bending test method was used according to the ASTM D198-22 (ASTM, 2022) with 1500 mm clear span. The four point bending test method consists of two load points and two support points. With the concept of two load points as shown in Figure 1, empirical data of the force in the pure bending moment can be obtained in the load span section, which is an important parameter for calculating the normal bending stress.

The test specimens of glue-laminated beams and bolt-laminated wood consisted of 4 laminae 60mm x 40mm (lamina thickness is 40mm), so the beam size was 60mm x 160mm. In bolted-laminated beams, the spacing between bolts was 100mm with 10mm bolt's diameter. The function of bolts is to prevent horizontal sliding due to internal forces, namely shear forces.

The number of test objects was 3 (three) for bolt-laminated beams and 3 (three) for glue-laminated beams. All test objects were made in dry conditions (moisture content ranging from 12% to 16%). The flexural strength referred to in this research is the flexural stress calculated under proportional load condition (F_b), while the MoR is the flexural stress calculated under ultimate or peak load.

Flexural Tests

Flexural testing for all specimens are carried out using a Universal Testing Machine (UTM) HT-9501 Electro-Hydraulic Servo (maximum load capacity 1000 kN) with output data in the form of a history curve of the relationship between flexural load and mid-span of beam deformation. Figure 1 shows the test equipment that used in the research.

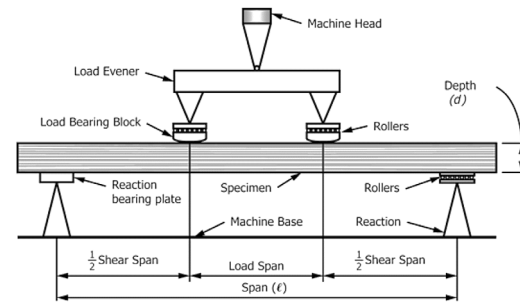


Figure 1. The flexural test in accordance with ASTM D198-22 (ASTM, 2022)

The general equation of normal (flexural) stress (Hibbeler, 2023) can be solved using Equation 1a.

$$\sigma = \frac{M \cdot y}{I_x} \quad (1.a)$$

$$I_x = \frac{1}{12} b \cdot d^3 \quad (1.b)$$

Flexural stress is calculated using Equation 2 (Goodno and Gere, 2021). Flexural strength is one of a material properties of a beam, which is the stress that occurs in a material before it yields in a flexural test using destructive method. In term of yield or proportional point,

$$F_b = \sigma \quad (2)$$

$$M = P_y \cdot a \quad (3)$$

where σ is normal (flexural) stress, F_b is flexural strength (MPa), M is moment at yield point (N.mm), P_y is yield point (N), a is length of $\frac{1}{2}$ shear span (meter) or 500mm in this research (see Figure 1), y is distance from surface area of cross-section to center of cross-section (meter), I_x is the cross-section moment of inertia (m^4), b is the beam width (mm), and d is the beam thickness (mm).

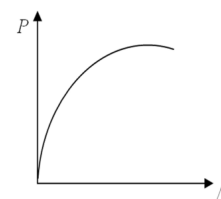


Figure 2. Flexural Load vs Mid-span deformation curve.

Figure 2 shows a schematic history of the flexural load vs mid-span of beam deformation relationship curve obtained from experimental test results. Modulus of Rupture (MoR) is a material property, defined as the stress in a material just in term of ultimate or peak load in a destructive method which is flexure test. MoR represents the ultimate stress experienced within the material at its moment of collapse of specimen.

$$MoR = \frac{M_{ult} \cdot y}{I_x} \quad (4)$$

where MoR is modulus of rupture and M_{ult} is moment at ultimate point (N.m).

Determining The Proportional or Yield Load

In this study, the determination of proportional or yield load uses the reference of SIA 265 Code (SIA, 2003). This is because most of beam members are not able to develop mechanisms of full plastic at failure.

In this study, the definitions of ductility ratio, as a ratio between an ultimate or peak deformation divided by yield or proportional deformation (Jorissen and Massimo, 2011), as shown in Figure 3. The ductility ratio (μ) of beam can be calculated using Equation 5.

$$\mu = \frac{\Delta_u}{\Delta_y} \quad (5)$$

where Δ_y is the displacement in term of proportional or yield load and Δ_u is the displacement in term of ultimate or peak load.

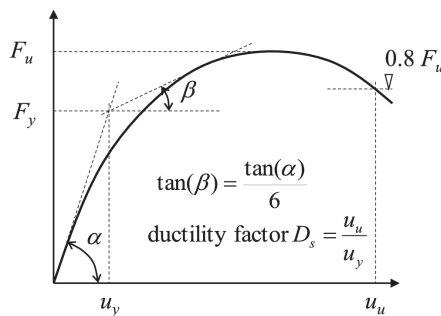


Figure 3. Determining the yield or proportional deformation (u_y) according to SIA 265 (SIA, 2003).

Results and Discussion

Figure 4 shows the bending test of a bolt-laminated beams in the laboratory. The specimen has a rectangular cross-section with a width of 60 mm (b) and a height of 160 mm (d), not included in the slender category, so that in testing up to the ultimate or peak load, no buckling and no horizontal deformation occurred in the middle of the span. The test results, namely the beam failure pattern, are shown in Figure 5 for the bolt-laminated beam specimens and Figure 6 for the glue-laminated beam specimens.



Figure 4. Flexural testing of specimens according to ASTM D198-22 (ASTM, 2022)



Figure 5. Bolt-laminated specimens after flexural tests



Figure 6. Glue-laminated specimens after flexural tests

The test results of all laminated-bolted beams generally consist of simple tension failures that occur in laminates that experience tensile stress. Meanwhile, for the glue-laminated beam test specimens, the failures that occurred were simple tension on first specimen and in the glue part on second and third specimens.

Figure 7 shows the test results of the bending load vs deflection curve of the glue-laminated beams. While Figure 8 shows the test results of the bending load vs deflection curve of the bolt-laminated beams. However, bolt-laminated beams has a smaller stiffness than glue-laminated beams, but exhibits more ductile behavior.

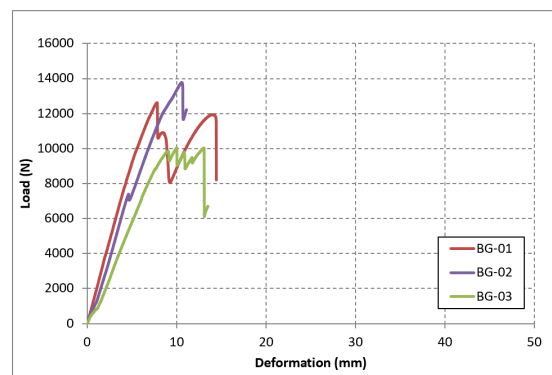


Figure 7. Flexural load vs mid-span of beam deformation curve obtained from glue-laminated timber specimens

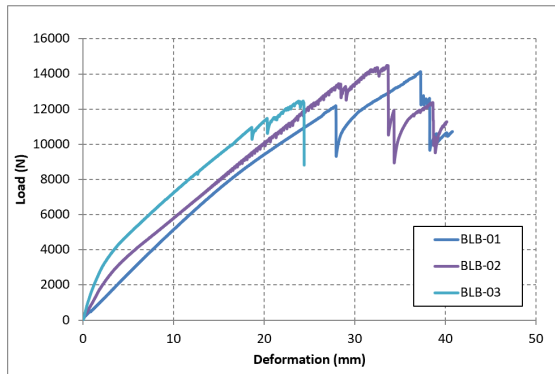


Figure 8. Flexural Load vs Mid-span of beam Deformation Curve obtained from bolt-laminated timber specimens

Table 2. Tests results: Load, Deformation, and Ductility ratio of bolt-laminated timber beams (BLB).

Spec.	P_y (kN)	Δ_y (mm)	P_u (kN)	Δ_u (mm)	μ
BLB-01	10.87	18.43	12.28	24.32	1.32
BLB-02	12.33	25.97	14.32	33.58	1.29
BLB-03	12.19	27.30	14.12	37.24	1.36
Mean	11.80	23.90	13.57	31.71	1.33

Table 3. Tests results: Load, Deformation, and Ductility ratio of glue-laminated timber beams (BG).

Spec.	P_y (kN)	Δ_y (mm)	P_u (kN)	Δ_u (mm)	μ
BG-01	11.78	6.96	12.59	7.83	1.13
BG-02	13.12	9.82	13.75	10.64	1.08
BG-03	9.40	8.33	10.04	10.03	1.20
Mean	11.43	8.37	12.13	9.50	1.14

Table 2 and Table 3 shows the calculation results, namely load at proportional limit conditions (P_y), load at ultimate limit or pak conditions (P_{ult}), deformation at proportional conditions (Δ_y), deformation at peak conditions (Δ_u), and ductility ratio (μ) which was calculated using Equation 5.

Table 4. Results of Flexural Strength and Modulus of Rupture of bolt-laminated timber beams.

Specimen	M_y (kN.m)	F_b (MPa)	M_{ult} (kN.m)	MoR (MPa)
BLB-01	2.72	10.62	3.07	11.99
BLB-02	3.08	12.04	3.58	13.99
BLB-03	3.05	11.90	3.53	13.79
Mean	2.95	11.52	3.39	13.26

Table 5. Results of Flexural Strength and Modulus of Rupture of glue-laminated timber beams.

Specimen	M_y (kN.m)	F_b (MPa)	M_{ult} (kN.m)	MoR (MPa)
BG-01	2.94	11.50	3.15	12.30
BG-02	3.28	12.81	3.44	13.43
BG-03	2.35	9.18	2.51	9.80
Mean	2.86	11.16	3.03	11.84

Table 4 and Table 5 shows the results of the calculation of flexural strength (F_b) which was calculated using Equation 2 and the modulus of rupture (MoR) which was calculated using Equation 4. Proportional limit or yield load (P_y) is the condition when a change occurs from an elastic to an inelastic condition.

The calculation results of ductility ratio (μ) as shows in Table 2 and Table 3 indicated that bolts as mechanical connection devices, namely horizontal shear restraints on laminated beams, provide an impact, namely more ductile behavior than laminated beams bonded with glue. Glue is, however, a brittle material.

Laminated-bolt beams have a flexural strength (F_b) of 11.52 MPa or 17.71% lower than solid beams (Chauf, 2019), while laminated-glued beams have a flexural strength of 11.16 MPa or 20.28% lower than solid beams. This is the impact of the influence of slip between laminae when the load acts on the beam. However, the laminated cross-section will not be the same as the solid cross-section.

Conclusions

Results obtained from this study indicated that the flexural strength of bolt-laminated and glue-laminated Sengon (*Albizia falcataria*) timber beams is not significantly different. The results of the study were that the flexural strength of bolt-laminated beams were 3.1% higher compared to the flexural strength of glue-laminated beams.

The modulus of rupture of bolt-laminated beams were 8.4% higher compared to the modulus of rupture of glue-laminated beams. The ductility of bolt-laminated beams were 14.2% higher compared to glue-laminated beams. These results indicate that the glue has an impact, namely the beam has brittle behavior or limited ductility, while the bolts have an impact, namely the beam behaves more ductile.

The general conclusion is that mechanical laminated timber technology can be an alternative to produce beams with larger cross-sectional sizes compared to solid timber, especially for low-grade wood, so that it can be used as part of the structural elements of buildings.

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A Study on the Application of Wax on Linden (*Tilia tomentosa* Moench.) Wood

Hüseyin Peker, Göksel ULAY, & Ümit Ayata

Abstract

Linden wood is used in match production, double bass making, carving, musical instrument crafting, and furniture production. This study examined the effects of applying varying numbers of wax coats (1, 2, and 3 coats) on the color parameters, glossiness values, and whiteness index (W^*) values of linden (*Tilia tomentosa* Moench.) wood surfaces. A comparison was made between untreated surfaces and those treated with three different wax applications. According to the obtained results, all tests except for hue angle were found to be significant in the analysis of variance. The ΔE^* values were found to be 5.13 for the application of 1 coat of wax, 6.53 for the application of 2 coats of wax, and 6.95 for the application of 3 coats of wax. After applying waxes to the wood, reductions were noted in W^* and L^* values, while enhancements were detected in glossiness readings at 60 and 85 degrees, as well as in a^* , C^* , h° , and b^* values.

Keywords: Wax, color, linden, whiteness index, glossiness

Introduction

In English, the word “wax” refers to a solid substance that melts with heat, typically smooth and shiny. This term is often used to describe materials such as wax, beeswax, or polish. It is commonly used to refer to substances used in furniture polish, car wax, or candle making. Additionally, the term “wax” is also used to denote the wax used in the music recording industry for duplicating or preserving recordings. (Bulian and Graystone 2009).

Most wax polishes consist of blends of different grades of waxes, both natural and synthetic resins, and additional components to achieve specific desired properties. These ingredients are typically dissolved or dispersed in an appropriate solvent or dispersing medium (Roberts 1968).

The layer of plant wax comprises a complex mixture of various classes of compounds, including fatty acids and long-chain n-alkanes. Among these, the long-chain n-alkanes are particularly valuable biomarkers because of their high resistance to natural degradation (Eglinton and Eglinton 2008).

Due to increasing environmental awareness among consumers and the avoidance of wood treated with biocidal processes and unsustainable tropical forests, the importance of wax use in the wood preservation industry, especially in Europe, is increasing. Therefore, the industry is interested in developing alternatives such as treatments with waxes (Lesar *et al.* 2011).

The impregnation depth of wood depends on the viscosity of the wax and the porosity of the wood. After filling the cell voids, waxes can reduce hygroscopicity and water absorption (Papadopoulos and Pougioula 2010).

High gloss is predominantly achieved from waxes through a migration mechanism. Waxes tend to fill micro voids that form as the coating dries, providing a smooth, continuous film. Typically, only small amounts of waxes

(0.5% to 2% solids) are needed to enhance gloss (Bower 2005).

The composition of crude oils includes several bipolar species capable of forming emulsions, and variations in the polar and non-polar parts of these species determine the diversity of emulsion sizes. Differences in chemical functionality (e.g., heterocyclic amines, sulfurs, oxides, carboxyl, poly- and mono-aromatic, alkane and alkene groups, etc.) and, consequently, variations in emulsion stabilities, are therefore prone to be extensive (Becker 1997).

Various types of beeswax and wax-containing chemicals prepared on different types of wood surfaces have been reported in the literature [beeswax and Manchurian ash (*Fraxinus mandshurica* Rupr.) wood (Niu and Song 2021), Ebony Macassar (*Diospyros celebica* Bakh.) (Kaplan *et al.* 2024), Siberian pine (*Pinus sibirica*) (Çamlıbel and Ayata, 2024a), ebony Afrika (*Diospyros crassiflora* Hiern.) (Çamlıbel and Ayata, 2024b), lemon (*Citrus limon* (L.) Burm.) (Çamlıbel and Ayata, 2024c), beeswax and poplar (*Populus ussuriensis* Kom) wood (Ren *et al.* 2016), wax and European walnut (*Juglans regia*) and sycamore maple (*Acer pseudoplatanus*) wood (Liu *et al.* 2022), beeswax and European cherry (*Prunus avium* L) and spruce (*Picea abies* (L) Karst) wood (Petric *et al.* 2004), polypropylene wax and Japanese elm (*Ulmus davidiana* Planch var. japonica) wood (Wang and Song 2022), beeswax and poplar (*Populus alba* L.), lime (*Tilia grandifolia* Ehrh.), Scots pine (*Pinus sylvestris* L.), and oriental beech (*Fagus orientalis* L.) wood (Akçay 2020), propolis and Scots pine (*Pinus sylvestris* L.), Nordmann fir (*Abies nordmanniana*), oriental spruce (*Picea orientalis* L.) wood (Akçay *et al.* 2022)].

Linden wood is used in the production of drawing boards and picture frames, carving, turning, and modeling, as well as for hat molds, boxes, chests, beehives, plywood, blockboard, and primer coating areas (Hammond *et al.* 1969).

In the literature, it has been observed that wax application on the surfaces of linden wood used in wood processing industries has not been conducted. This study was conceived to address this gap. Additionally, testing wax application is important for introducing a new perspective on the usage areas of linden wood.

In this study, wax applications of different coat numbers were tested on linden (*Tilia tomentosa* Moench.) wood, and whether there were differences between layers was examined through various surface tests.

Materials and Methods

Wood Material

Linden (*Tilia tomentosa* Moench.) wood samples were prepared in dimensions of 100 x 200 x 20 mm according to the TS ISO 13061-1 (2021) standard.

Wax

In the study, an oil mixture containing a combination of natural and synthetic waxes (appearance: paste, color: neutral, odor: characteristic, solubility in water: dispersible but insoluble, dry residue: 30%, and pH value: 7.6) was used. The wax was purchased from a specialized wood preservative supplier.

Application of Wax on Wooden Surfaces

The oil mixture containing a combination of natural and synthetic waxes was applied to wooden surfaces using a brush, in 1, 2, and 3 layers.

Determination of Some Surface Properties

Glossiness tests (ISO 2813 1994) (ETB-0833 model gloss meter), whiteness index (W^*) values (ASTM E313-15e1 2015) (Whiteness Meter BDY-1), and color changes (ASTM D 2244-3 2007) (CS-10) were measured. Total color differences were calculated using the following formulas.

$$\Delta L^* = (L^*_{\text{wax applied sample}} - L^*_{\text{control}}) \quad (1)$$

$$C^* = [(a^*)^2 + (b^*)^2]^{0.5} \quad (2)$$

$$h^\circ = \arctan(b^*/a^*) \quad (3)$$

$$\Delta H^* = [(\Delta E^*)^2 - (\Delta L^*)^2 - (\Delta C^*)^2]^{0.5} \quad (4)$$

$$\Delta C^* = (C^*_{\text{wax applied sample}} - C^*_{\text{control}}) \quad (5)$$

$$\Delta a^* = (a^*_{\text{wax applied sample}} - a^*_{\text{control}}) \quad (6)$$

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{0.5} \quad (7)$$

$$\Delta b^* = (b^*_{\text{wax applied sample}} - b^*_{\text{control}}) \quad (8)$$

ΔC^* : chroma difference or saturation difference, and ΔH^* : hue difference or shade difference are indicated. Table 1 provides important information on other parameters (Lange, 1999). ΔE^* comparison criteria (DIN 5033 1979) are given in Table 2.

Table 1. Information regarding Δa^* , ΔL^* , Δb^* , and ΔC^* values (Lange 1999)

Test	Negative Description	Positive Description
ΔC^*	More dull, matte than the reference	Clearer, brighter than the reference
ΔL^*	Darker than the reference	Lighter than the reference
Δb^*	More blue than the reference	More yellow than the reference
Δa^*	More green than the reference	More red than the reference

Table 2. Comparison criteria for ΔE^* assessment (DIN 5033 1979)

Visual	Total Colour Difference	Visual	Total Colour Difference
Undetectable	<0.2	Distinct	1.5 - 3.0
Very Weak	0.2 - 0.5	Very Distinct	3.0 - 6.0
Weak	0.5 - 1.5	Strong	6.0 - 12.0
		Very Strong	> 12.0

Statistical Analysis

A statistical program was utilized to calculate standard deviations, variance analysis results, means, minimum and maximum values, homogeneity groups, and percentages (%) of changes.

Results and Discussion

According to the variance analysis results presented in Table 3, it is observed that the applied wax coat number factor is insignificant for the hue angle (h°) value, while for all other tests, the applied wax coat number factor is found to be significant (Table 3).

Table 3. Analysis of variance results (*: Significant)

Test	Sum of Squares	df	Mean Square	F value	Sig.
L^*	68.344	3	22.781	890.208	0.000*
a^*	16.627	3	5.542	196.854	0.000*
b^*	225.323	3	75.108	623.424	0.000*
C^*	241.858	3	80.619	626.260	0.000*
h°	0.716	3	0.239	1.767	0.171**
$\perp 20^\circ$ glossiness	2.167	3	0.722	86.382	0.000*
$\perp 60^\circ$ glossiness	64.871	3	21.624	254.978	0.000*
$\perp 85^\circ$ glossiness	248.850	3	82.950	1013.646	0.000*
$\parallel 20^\circ$ glossiness	0.897	3	0.299	176.410	0.000*
$\parallel 60^\circ$ glossiness	47.715	3	15.905	324.406	0.000*
$\parallel 85^\circ$ glossiness	465.321	3	155.107	341.123	0.000*
W^* (\perp)	489.280	3	163.093	1405.977	0.000*
W^* (\parallel)	748.181	3	249.394	1105.959	0.000*

Table 4 presents the results for the color parameters. For the L^* value, the highest value was observed in the control experiment samples (78.45), while the lowest was obtained in the samples treated with 3 layers of wax (75.11). For L^* , decreases of 2.21%, 3.80%, and 4.26% were observed with 1, 2, and 3 layers of wax, respectively. It is determined that increases in the number of layers result in decreases in this parameter (Table 4).

The a^* parameter exhibited its lowest value in the control experiment group samples at 4.96, while the highest value was recorded in samples treated with three layers of wax, reaching 6.56. With the application of 1, 2, and 3 layers of wax, increases of 25.60%, 30.85%, and 32.26% were observed, respectively. This indicates a consistent trend of increasing values with each additional layer of wax, as indicated in Table 4.

In relation to the b^* value, the control experiment group samples exhibited the lowest measurement (16.81), whereas the samples treated with 3 layers of wax demonstrated the highest value (22.68). Incremental increases of 27.66%, 33.31%, and 34.92% were noted with 1, 2, and 3 layers of wax, respectively. The trend indicates that as the number of wax layers applied increases, so does this parameter (as depicted in Table 4).

In the case of the C^* value, the control experiment group samples exhibited the lowest value (17.52), whereas

the samples treated with 3 layers of wax demonstrated the highest value (23.61). This resulted in increases of 27.57%, 33.16%, and 34.76% with 1, 2, and 3 layers of wax, respectively. The trend indicates that as the number of layers increases, so do the values of this parameter (Table 4).

In terms of the h° value, the control group samples exhibited the lowest measurement at 73.54, whereas the highest reading was recorded in samples treated with three layers of wax, reaching 73.88. This indicates an incremental trend of 0.37%, 0.42%, and 0.46% with each successive layer of wax 1, 2, and 3 layers, respectively. These findings illustrate a proportional rise in values as the number of wax layers increases, as outlined in Table 4.

Akçay (2020) reported a decrease in L^* values and an increase in a^* and b^* values with the application of wax to linden, beech, poplar, and Scots pine woods, while Liu *et al.* (2022) reported similar results for European walnut and sycamore maple woods. These findings are consistent with the results obtained in our study.

Additionally, in the literature, studies have reported changes in color, glossiness, and whiteness index values when chemicals derived from waxes were applied to different wood material surfaces (Çamlıbel and Ayata, 2024a; b; c; Kaplan *et al.* 2024).

Table 4 Results of color parameters

Test	Wax Application	N	Mean	Change (%)	HG	Standard Deviation	Minimum	Maximum	COV
L^*	Control	10	78.45	-	A*	0.14	78.24	78.72	0.18
	1 layer	10	76.72	↓2.21	B	0.17	76.50	77.01	0.22
	2 layers	10	75.47	↓3.80	C	0.14	75.22	75.66	0.18
	3 layers	10	75.11	↓4.26	D**	0.19	74.80	75.41	0.25
a^*	Control	10	4.96	-	C**	0.16	4.72	5.19	3.25
	1 layer	10	6.23	↑25.60	B	0.15	5.98	6.51	2.38
	2 layers	10	6.49	↑30.85	A	0.16	6.26	6.80	2.41
	3 layers	10	6.56	↑32.26	A*	0.20	6.27	6.81	3.05
b^*	Control	10	16.81	-	C**	0.44	16.04	17.30	2.63
	1 layer	10	21.46	↑27.66	B	0.20	21.21	21.88	0.91
	2 layers	10	22.41	↑33.31	A	0.41	21.87	23.13	1.84
	3 layers	10	22.68	↑34.92	A*	0.28	22.32	23.12	1.23

C*	Control	10	17.52	-	C**	0.45	16.79	18.00	2.60
	1 layer	10	22.35	↑27.57	B	0.21	22.10	22.77	0.96
	2 layers	10	23.33	↑33.16	A	0.41	22.81	24.04	1.74
	3 layers	10	23.61	↑34.76	A*	0.31	23.21	24.09	1.32
h°	Control	10	73.54	-	A**	0.39	72.87	74.03	0.53
	1 layer	10	73.81	↑0.37	A	0.31	73.29	74.30	0.42
	2 layers	10	73.85	↑0.42	A	0.41	73.27	74.55	0.56
	3 layers	10	73.88	↑0.46	A*	0.36	73.31	74.43	0.48

HG: Homogeneity Group, COV: Coefficient of Variation, N: Number of Measurements, *: Highest value, **: Lowest value

Table 5 shows the results of the glossiness values. Glossiness values at 60 and 85 degrees made in perpendicular and parallel directions showed an increase with the application of wax layers. In addition, decreases

were obtained with 2 and 3 layers of wax applications for glossiness values made in both directions at 20 degrees (Table 5).

Table 5 Results of glossiness values

Test	Wax Application	N	Mean	Change (%)	HG	Standard Deviation	Minimum	Maximum	COV
⊥ 20°	Control	10	0.60	-	B**	0.00	0.60	0.60	0.00
	1 layer	10	0.60	0.00	B**	0.00	0.60	0.60	0.00
	2 layers	10	1.08	↑80.00	A*	0.04	1.00	1.10	3.90
	3 layers	10	1.05	↑75.00	A	0.18	0.90	1.30	16.95
⊥ 60°	Control	10	2.05	-	C**	0.33	1.70	2.50	16.14
	1 layer	10	3.64	↑77.56	B	0.12	3.50	3.80	3.22
	2 layers	10	5.16	↑151.71	A*	0.21	4.80	5.30	4.00
	3 layers	10	5.10	↑148.78	A	0.42	4.80	5.70	8.16
⊥ 85°	Control	10	0.22	-	D**	0.13	0.10	0.40	59.84
	1 layer	10	1.03	↑368.18	C	0.20	0.80	1.30	19.44
	2 layers	10	5.20	↑2263.64	B	0.19	5.00	5.50	3.63
	3 layers	10	5.91	↑2586.36	A*	0.48	5.40	6.50	8.19
∥ 20°	Control	10	0.62	-	C	0.04	0.60	0.70	6.80
	1 layer	10	0.60	↓3.23	C**	0.00	0.60	0.60	0.00
	2 layers	10	0.94	↑51.61	A*	0.05	0.90	1.00	5.49
	3 layers	10	0.87	↑40.32	B	0.05	0.80	0.90	5.55
∥ 60°	Control	10	2.18	-	D**	0.32	1.80	2.70	14.79
	1 layer	10	3.84	↑76.15	C	0.18	3.60	4.00	4.79
	2 layers	10	4.96	↑127.52	A*	0.13	4.90	5.20	2.55
	3 layers	10	4.73	↑116.97	B	0.21	4.50	5.00	4.35
∥ 85°	Control	10	0.14	-	C**	0.08	0.10	0.30	60.23
	1 layer	10	1.74	↑1142.86	B	0.16	1.60	2.00	9.46
	2 layers	10	7.78	↑5457.14	A*	1.31	6.80	10.20	16.89
	3 layers	10	7.55	↑5292.86	A	0.24	7.20	7.70	3.20

HG: Homogeneity Group, COV: Coefficient of Variation, N: Number of Measurements, *: Highest value, **: Lowest value

Table 6 presents the results of the whiteness index (W^*) values. For W^* values, decreases were observed with all layer numbers in both directions. W^* values perpendicular to the fibers were found to be higher than parallel to the fiber results. Decrease rates for W^* values perpendicular to the fibers were determined to be 12.06%, 19.26%, and 21.98%

with 1, 2, and 3 layer applications, respectively, while decrease rates for W^* values parallel to the fibers were determined to be 24.10%, 26.03%, and 27.62% with 1, 2, and 3 layer applications, respectively. W^* values decreased with an increase in the number of layers in both directions (Table 6).

Table 6 Results of whiteness index (W^*) values

Test	Wax Application	N	Mean	Change (%)	HG	Standard Deviation	Minimum	Maximum	COV
W^* ⊥	Control	10	41.12	-	A*	0.19	40.80	41.30	0.47
	1 layer	10	36.16	↓12.06	B	0.16	35.90	36.30	0.44
	2 layers	10	33.20	↓19.26	C	0.12	33.10	33.40	0.35
	3 layers	10	32.08	↓21.98	D**	0.62	31.50	32.80	1.94

	Control	10	38.30	-	A*	0.94	37.30	39.60	2.44
W [*]	1 layer	10	29.07	↓24.10	B	0.09	28.90	29.20	0.33
	2 layers	10	28.33	↓26.03	C	0.08	28.20	28.40	0.29
	3 layers	10	27.72	↓27.62	D**	0.10	27.60	27.90	0.37

HG: Homogeneity Group, COV: Coefficient of Variation, N: Number of Measurements, *: Highest value, **: Lowest value

Literature reports indicate that surface properties of wooden materials change with the application of waxes to various wood species such as olive (*Olea europaea* L.) (Peker *et al.* 2024a), balau red (*Shorea guiso*) (Peker *et al.* 2024b), American walnut (*Juglans nigra* L.) (Ayata 2024), American white oak (*Quercus alba*) (Ayata and Ayata 2024), American black cherry (*Prunus serotina*) (Ayata and Çamlıbel 2024), magnolia (*Magnolia grandiflora* L.) (Ayata *et al.* 2024), and plum (*Prunus domestica* L.) (Peker *et al.* 2024c), among others.

Table 7 shows the results of total color differences. ΔE^* values were found to be 5.13 with 1 layer of wax application, 6.53 with 2 layers of wax application, and 6.95

with 3 layers of wax application. ΔL^* values were determined to be negative for all layer numbers, while Δa^* , Δb^* , and ΔC^* values were obtained positively (Table 7).

ΔH^* values were determined to be 0.12 with 1 layer of wax application, 0.10 with 2 layers of wax application, and 0.11 with 3 layers of wax application, and the values were found to be very close to each other (Table 7). When compared with the values given in the color change criteria (DIN 5033, 1979), “very pronounced (3.0 to 6.0)” results were obtained with 1 layer of wax application, and “strong (6.0 to 12.0)” results were obtained with 2 and 3 layer wax applications (Table 7).

Table 7. Results of total color differences

Wax Application	ΔL^*	Δa^*	Δb^*	ΔC^*	ΔH^*	ΔE^*	The color change criteria (DIN 5033, 1979)
1 layer	-1.72	1.27	4.66	4.83	0.12	5.13	Very distinct (3.0 - 6.0)
2 layers	-2.98	1.53	5.61	5.81	0.10	6.53	Strong (6.0 - 12.0)
3 layers	-3.34	1.59	5.88	6.09	0.11	6.95	

Conclusions

Following the application of waxes to wood, decreases were observed in the W^* and L^* values, while increases were obtained in the C^* , a^* , h° , and b^* values, as well as in the glossiness values at 60 and 85 degrees. It is recommended to conduct a wetting test using different chemicals on the obtained materials. Considering the total color difference results obtained, it can be said that applying wax in two coats will be sufficient for linden wood.

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Chemical Components of Boiling-Treated Sengon (*Paraserianthes falcataria* (L.) I. C. Nielsen) Wood

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Abstract

Sengon is a fast-growing wood species widely distributed on Java Island and commonly utilised for plywood production. It is frequently subjected to boiling treatment to reduce veneer defects, making it important to understand how this treatment affects its properties. This study investigated the effect of boiling treatment on the properties of sengon wood, involving three seven-year-old trees which were subjected to a control and an experimental treatment at 80°C for ten hours. The wood specimens were divided into near-pith, middle, and near-bark sections. The specimens were powdered to a size of 40–60 mesh for successive extraction with n-hexane, methanol, and hot water. The total phenolic content was measured from the methanol-soluble extract, and the total polysaccharide content was measured from the hot-water-soluble extract. Chemical components such as holocellulose, alpha-cellulose, hemicellulose, lignin, ash, and silica contents, as well as pH values, were determined. T-test revealed that boiling treatment significantly reduced extractive levels (in ethanol-toluene and methanol solubles) but significantly increased total phenolic and polysaccharide contents. No significant effect was observed on cell wall chemical components. One-way ANOVA indicated that the radial direction significantly affected methanol-soluble extractive content, total phenolic content, and total polysaccharide content.

Keywords: Boiling treatment, radial direction, chemical properties, wood extractives, *Paraserianthes falcataria* (L.) I.C.Nielsen.

Introduction

Sengon (*Paraserianthes falcataria* (L.) I. C. Nielsen) is one of the most popular tree species, particularly prevalent in community forests on Java island. Known for its fast growth, it is capable of producing large volumes of wood under favorable conditions (Amelia *et al.* 2021). These fast growth and high yield make sengon wood the main raw material in the plywood industry (Alim and Suseno 2022).

Sengon wood, used as a plywood material, is prone to have physical defects following felling due to the high growth stress experienced by standing trees. These defects reduce the price or potential added value of the wood (Marsoem *et al.* 2014). Additionally, cracks in the logs can compromise the yield during the wood peeling process. Therefore, a special treatment to minimise these risks is necessary. For example, soaking wood in hot water at 80°C for 10 hours has been reported to reduce wood damage during peeling (Mazela *et al.* 2004). Technically, wood boiling affects not only the wood's physical properties but also its chemical properties.

Boiling treatment is believed to improve the quality of veneer by improving the gluing process through the reduction of extractive levels (Widyorini and Puspitasari 2009). Previous research on kamper and keruing woods showed that boiling treatment significantly reduced the extractive contents soluble in alcohol-benzene, hot water, and cold

water (Wahjudi 1990). However, the effect of boiling treatment on sengon wood properties has yet to be widely investigated. Thus, this research aimed to find out how boiling treatment could alter the chemical components of sengon wood.

Materials and Methods

Materials and extraction

Three seven-year-old sengon trees were harvested from a community forest in Kedung Pomahan Kulon Hamlet, Kemiri District, Purworejo Regency, Central Java, by cutting at the bottom part. The logs were treated with soaking and boiling at 80°C for 10 hours (Fig. 1). The temperature applied followed previous temperature application to reduce the tree growth stress (patent number IDS000005180). After boiling, the logs were air-dried for approximately four hours and cut into 5 cm thick discs from the end parts (Fig. 2). The samples were then drilled near the bark, at the center, and near the pith at the radial direction and grounded to produce a 40–60 mesh powder size. A comparison was carried out between treated and untreated wood samples (control).

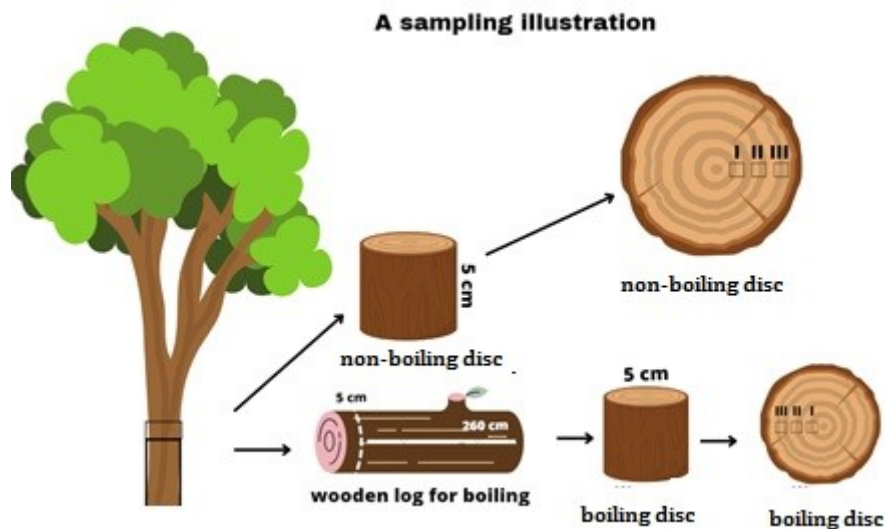


Figure 1. Scheme of wood sampling.



Figure 2. The boiling process of the 7-year-old sengon log

Determination of wood chemical components

Two extraction methods were applied to the sengon wood samples. The first method involved successive extraction of 2 g of dry wood using *n*-hexane, methanol, and hot-water solvents. The *n*-hexane and methanol extractions used a Soxhlet apparatus for six hours, while the hot-water extraction used a water bath at 100°C for approximately three hours. The extract filtrates were evaporated to obtain dry extracts. The *n*-hexane extractive content (HEC), methanol extractive content (MEC), and hot-water extractive content (WEC) were summed up to obtain the total extractive content (TEC). The second method involved successive extraction of 2 g of wood powder using ethanol-toluene solvent (ASTM D1107-96 and hot water (ASTM D 1110-96).

The wood extracts obtained using the first extraction method were analysed for total phenolic and polysaccharide contents, while the wood residue obtained using the second method was subjected to cell wall component determination. The total phenolic content was determined using the Folin-Ciocalteu method, with absorbance read at 765 nm using a UV-Vis spectrophotometer (WPA 800) (Gao *et al.* 2007). Meanwhile, the total polysaccharide content was determined in the hot-water-soluble extract (1000 ppm) using the phenol-sulfuric acid method, with absorbance measured at 490 nm (Dubois *et al.* 1956).

The extractive-free wood powder residue from the second method was used for determining the holocellulose, cellulose, and lignin contents. Holocellulose content was determined using the acid chlorite method (Browning 1967).

Cellulose content was determined using 17.5% NaOH immersion (Rowell 2005). Hemicellulose content was calculated as the difference between holocellulose and α -cellulose contents (Lukmandaru *et al.* 2018). Lignin content was determined using the Klason method, as specified by SNI 0492: 2008, involving a treatment with 72% sulfuric acid.

Ash and silica contents were measured based on SNI 14-1031-1989. The pH value was measured by soaking 1 g of wood powder in distilled water for 48 hours. After filtering, the pH value of the filtrate was measured using a pH meter (in duplicate).

Statistical analysis

The effects of boiling treatment factors on chemical components and extractive contents were analysed using t-test, while radial variation was analysed using one-way ANOVA (analysis of variance). Tukey's Honestly Significant Difference (HSD) was used for post-hoc analysis (Steel *et al.* 1997)

Results and Discussion

Chemical properties

Sengon wood is primarily composed of carbohydrates, including holocellulose (which consists of cellulose and hemicellulose) and pectin, as well as lignin. The primary components of sengon wood in this study are detailed in Table 1. The holocellulose content was within the range observed for sengon wood from West Kalimantan (Putra *et al.* 2018), whereas the α -cellulose content was higher (47.99–48.91%) than that observed for five-year-old sengon wood from West Java (Pari 1996). Furthermore, the sengon wood in this study had higher lignin content (26.58–30.81%) compared to the value reported for sengon wood from plantations in West Java (Pari and Hartoyo 1990). Based on three individual trees, the values of lignin content is within the range of 5-year-old sengon wood (Pari 1996). However, the range of lignin in this study is smaller than that of Windeisen *et al.* (2003) on teak wood from Panama (33.3–38.3%).

In this study, total extractive content was calculated by summing the extractive levels derived from successive extractions using *n*-hexane, methanol, and hot water. As presented in Table 3, the total extractive content in this study ranged from 5.23% to 11.57%, which was higher than those reported in other studies (Pari 1996; Martawijaya *et al.* 1989). The total phenolic content in the methanol-soluble extract in this study was higher than that of six- and eight-year-old Jati Unggul Nusantara wood, while the total polysaccharide content in the hot-water-soluble extract was lower (Rahman and Lukmandaru 2022).

Ash is an inorganic residue derived from various salts deposited in the cell wall and lumen (Sjostrom 1995). Silica, a major constituent of ash, is more varied in quantity in tropical broadleaf species (Shmulsky and Jones, 2011). In this research, the ash content ranged between 0.78% and

1.04%, which was lower than the 0.64% ash content found in five-year-old sengon wood and fell within the range of 0.37–0.50% for sengon wood from West Java plantations (Pari 1996; Pari and Hartoyo 1990). Ash content can be affected by the age of the wood, with higher ash content found in older wood (Pari and Saepuloh 2000). Additionally, silica content higher than 0.3% can accelerate saw blade dulling (Shmulsky and Jones 2011). In other words, the seven-year-old sengon wood in this study might negatively affect sawing tools as most samples had silica levels above 0.3%. Thus, a special treatment is needed to reduce the silica content.

The pH value of sengon wood in this study ranged from 5.83 to 6.06, which fell within the acidic range. This range is comparable to that found for mahogany wood (5.20–5.94) (Lukmandaru *et al.* 2018). Technically, a low pH value will accelerate the curing of UF adhesive in the gluing process during hot pressing for particleboard (Maloney 1993).

Boiling treatment

The t-test results provided in Table 2 showed that boiling had no significant effect on any of the wood cell wall components. It can be assumed that the carbohydrate components of the sengon wood in this study were not degraded, likely due to the low temperature used in the experiment. In contrast, Ates *et al.* (2009) demonstrated that heat treatment at 130°C for 2–8 hours had a significant effect on the degradation of wood carbohydrates in *Pinus brutia*. The finding of this study is in line with that of Ismanto and Saputro (2014), who found almost no change in cellulose content after boiling treatment of rubber wood. Similarly, lignin content did not indicate any significant difference after the boiling treatment due to the same reason.

Wood extractive contents were determined by two extraction methods. The t-test results showed that boiling treatment had no significant effect on the extractive contents soluble in hot water and *n*-hexane, and neither did it on the total extractives (Table 3). Although the extractive content soluble in *n*-hexane increased from 1.39% to 1.63% after boiling, the increase was not considered statistically significant. As *n*-hexane naturally dissolves terpene and fat groups, the lack of significant change in the extractive content in *n*-hexane indicates that these groups are not sensitive to boiling treatment at 80°C for 10 hours. In addition, although the average hot-water-soluble extractive content and total extractives decreased after boiling, these decreases were not significant. This is in contrast to an earlier study by Wahjudi (1990), which reported decreases in both hot-water-soluble and total extractive contents of kamper and keruing woods after boiling at 100°C.

Boiling significantly reduced the extractive contents soluble in ethanol-toluene and methanol, while it significantly increased the total phenolic and polysaccharide contents. After boiling, the extractive content soluble in ethanol-toluene decreased to 3.5%, compared to 4.36% in control. This pattern was also observed by Lukmandaru *et al.* (2018), who investigated the alcohol-benzene extractive content of

mahogany wood using the steaming method at temperatures of 90°C, 120°C, and 150°C for two hours. The observed reduction in extractive content might be due to the loss of volatile substances during heat treatment. Previous research reported that volatile substances were lost from *Pinus pinaster* wood as other components were degraded after heat treatment at 260°C (Bourgeois and Guyonnet 1988). Ates *et al.* (2009) also observed that extractives, mostly volatile compounds, were lost during heat treatment. Methanol

extractive content decreased from 3.97% to 2.99% after boiling, a more pronounced change compared to that reported for hot water, which is a relatively more polar solvent than methanol. Methanol technically dissolves various compounds in wood, such as flavonoids, tannins, carotenoids, and other phenol compounds. This suggests that boiling treatment has a more significant impact on wood phenolic components with lower molecular weight.

Table 1. Comparison of chemical components of sengon wood with previous studies.

Chemical properties	This research	Previous research results		
		Putra <i>et al.</i> (2018)	Pari & Hartoyo (1990)	Pari (1996)
Holocellulose (%)	74.86-76.21	69.16-88.33	59.41	70.9
α -cellulose (%)	47.99-48.91	57.11-74.21	46.31	46.62
Hemicellulose (%)	26.84-27.30	-	-	-
Lignin (%)	26.58-30.81	16.69-23.77	25.14	29.1
Ash (%)	0.78-1.04	-	0.81	0.64
Silica (%)	0.37-0.50	-	0.13	0.5
pH	5.83-6.01	-	-	-
Total extractive content (%)	5.23-6.01	-	5.39 ¹	2.06-4.34 ¹

Note: (a) Putra *et al.* (2018) (sengon wood from West Kalimantan with a diameter of 33 cm), (b) Pari & Hartoyo (1990) (sengon wood from West Java plantations), (c) Pari (1996) (5 year old sengon wood from West Java), (1) : Ethanol-benzene extractives.

Table 2. Average value of chemical components of sengon wood

Chemical properties	Control	Boiling
Holocellulose (%)	76.07	74.73 ^{NS}
α -cellulose (%)	48.72	48.06 ^{NS}
Hemicellulose (%)	27.35	26.67 ^{NS}
Lignin (%)	29.63	28.19 ^{NS}
Ash (%)	1.04	0.75 ^{NS}
Silica (%)	0.61	0.26 ^{NS}

Note: NS: Not significant in t-test.

Table 3. Average value of extractive content and pH value of sengon wood

Chemical properties	Obtained values		Treatment	
	Minimum	Maximum	Control	Boiling
Ethanol-toluene content (%)	2.68	4.92	13.09	10.5*
Hot water extractive content (%)	2.51	2.8	8.79	7.2 ^{NS}
<i>n</i> -heksane extractive content (%)	0.25	2.84	4.17	4.89 ^{NS}
Methanol extractive content (%)	2.1	8.59	11.92	8.97*
Hot water extractive content (%)	1.71	4.25	11.92	7.06 ^{NS}
Total extractive content (%)	5.23	11.57	23.2	20.9 ^{NS}
Total phenolic content (gallic acid equ./g sample)	16.01	277.05	259.34	421.52*
Total polysaccharide content (glucose equ./g sample)	43.96	155.16	149.988	337.76*
pH values	5.83	6.01	5.97	5.88 ^{NS}

Note: NS: Not significant in t-test, (*): highly significant in t-test at the 5% test level

The amount of total phenolics content increased from 86.45 (GAE/g sample) to 140.51 (GAE/g sample) after boiling. It is assumed that lignin compounds are degraded in the boiling process. However, no significant effect of boiling was found in lignin content (Table 2). It is assumed that the boiling factor with a temperature of 80 °C did not cause lignin

degradation. Hemicellulose degradation starts at 200-260°C while cellulose at 240-350 °C and lignin at a wider temperature range (280-500 °C) (Nawawi *et al.* 2018). The increase of the total phenolic content might be due to the degradation of phenol constituents in the form of phenolic acids contained in the hydrophilic part of wood extractives.

This trend was also observed on reeds boiling (Dhyanaputri *et al.* 2022). Another work (Tan *et al.*, 2014) showed that water can be used effectively to extract phenolic compounds in bitter melon where the optimal condition for water extraction by a single extraction at 80 °C for 5 minutes. Phenolic compounds are known to be sensitive to heating even though in a short time treatment (Ismail 2004),. The heat-sensitive nature could have a different effect, both increasing and decreasing the total phenolic content.

Total polysaccharide content increased from 50 GAE/g to 112.59 GAE/g sample after boiling treatment. In this study, cell wall components such as cellulose, hemicellulose, holocellulose, and lignin did not give significant differences after boiling treatment (Table 2). This suggests that the increase in total polysaccharide content after boiling is not caused by the degradation of wood cell wall components. Theoretically, softwood consists of galactoglucomannan (about 20%), arabinoglucuronoxylan (5%-10%), arabinogalactan, and other small amounts of polysaccharides (pectin and starch compounds) (Sjostrom 1998). Therefore, the increase in total polysaccharide content is probably due to the degradation of other components of small polysaccharides such as pectin and starch compounds. Starch is a polysaccharide in the form of a polymer of α -D-glucose (Erika 2010). Starch consists of two fractions that can be separated with hot water. The soluble fraction is called amylopectin and the insoluble fraction is called amylose (Hee-Young 2005). Generally, starch contains 15-30% amylose, 70-85% amylopectin, and 5-10% intermediate materials (Zulaidah 2012). The starch fractions dissolved in

this hot water during boiling caused the high total polysaccharide content.

Radial direction

One-way ANOVA results showed that radial direction had no significant effect on any of the wood cell wall components, including alpha-cellulose, holocellulose, hemicellulose, lignin, silica, and ash contents. This finding contrasts with that of Jahan *et al.* (2019), who investigated the effect of radial direction on ten-year-old *A. auriculiformis*. In addition, the pH value was found to be related to ash content, particularly in terms of the alkaline properties of its main components: Ca, Mg, and K (Panshin and de Zeeuw 1981). However, pH value does not fluctuate along radial direction. The low pH value near the pith or heartwood was generally due to the presence of phenolic compounds, which have weak acidic properties (Krilov and Lasander 1998). The pH value observation revealed that ash and phenolics may not be the only factors influencing pH value.

These one-way ANOVA results further indicated that radial direction had no significant effect on the extractive contents soluble in hot water, *n*-hexane, and methanol, as well as the total extractives. This suggests that sengon wood has a relatively uniform extractive composition along the radial direction. This absence of a significant effect might also be attributable to the unclear division between the heartwood and sapwood. In general, sengon wood has white heartwood and white sapwood, which makes it difficult to visually distinguish them (Martawijaya *et al.* 1989).

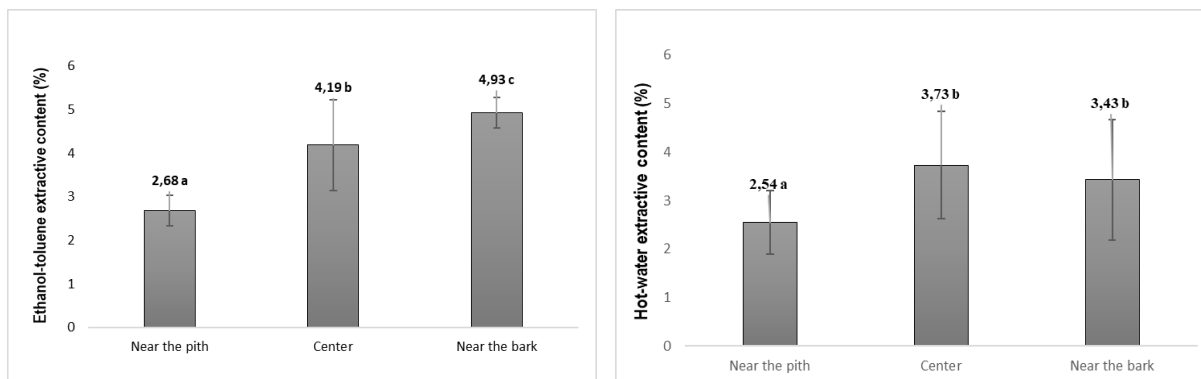


Figure 3. Radial direction of ethanol-toluene extractive content and hot-water extractive content in sengon wood. The same letters on the same graphic are not statistically different at $p < 0.05$ by Tukey's test.

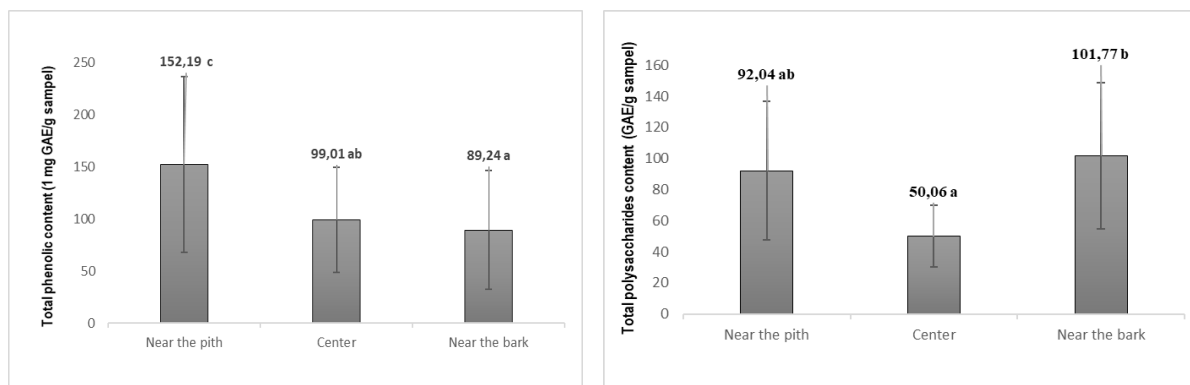


Figure 4. Radial direction of total phenolic and total polysaccharide contents in sengon wood. The same letters on the same graphic are not statistically different at $p < 0.05$ by Tukey's test.

Radial direction had significant effects on the extractive contents soluble in toluene-ethanol and hot water, as well as the total phenolic and total polysaccharide contents. Tukey's test showed the highest ethanol-toluene-soluble extractive content near the bark (Fig. 3), which is at odds with the finding of Shmulsky and Jones (2011). This result indicates a higher concentration of non-polar components in this part of the wood. It is likely that heartwood in seven-year-old sengon wood is not present near the pith. Heartwood formation is characterized by the conversion of polysaccharides from the sapwood into phenolics (Nobuchi *et al.* 2005). In addition, the lowest level of hot-water-soluble extractives was observed near the heartwood. This pattern is in agreement that substances soluble in water such as starch and pectin are more abundant in the sapwood near the bark than in the heartwood (Fengel and Wegener 1984). The highest average value of total phenolic content was found near the pith (152.19 GAE/g sample) (Fig. 4), which might be related to heartwood formation. Earlier work by Datta and Kumar (1987) on teak wood showed that the highest amounts of phenolics were found in the heartwood, which decreased towards the bark and were completely depleted in the outer sapwood. Similarly, Asdar *et al.* (2016) observed the highest ethanol-toluene-soluble extractive content in the heartwood of ebony wood, which tended to decrease towards the sapwood. This decrease was due to the accumulation of secondary metabolites, such as phenolics, in the cell wall and lumen of the heartwood.

The highest total polysaccharide content of 101.77 GAE/g was obtained near the bark. Tukey's HSD showed a significant difference between the center and the part near the bark. Similarly, the highest concentrations of hot-water-soluble extractives were found in the same region. The sapwood is generally richer in nutrients such as sucrose and wood glycosides, while the heartwood and bark are usually richer in secondary metabolites (Gao *et al.* 2007).

Conclusions

Boiling treatment had significant effects on methanol-soluble extractive content, total phenolic content, and total

polysaccharide content, but it did not have any significant effect on cell wall components. These trends may be attributable to the application of low temperatures in the experiment. Radial direction had significant effects on hot-water-soluble extractive content, total phenolic content, and total polysaccharide content at the 5% significance level. As with boiling treatment, radial direction also lacked a significant effect on cell wall components. The variation in extractive contents might be related to heartwood formation, which necessitates a further study to confirm this hypothesis.

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Extractive Content and Antioxidant Activity of Cajuput Bark (*Melaleuca cajuputi* Powell)

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Abstract

Both wood and non-wood industries generate large amounts of residues from processes like bark peeling and removal. In the cajuput oil industry, the waste products include distillation residues and cajuput bark. The diverse extractives and chemical components found in cajuput bark have the potential for enhanced value through various applications. This study aimed to determine the chemical components, extractive content (EC), and percentage inhibition of antioxidant activity (%IAA) of cajuput bark extract to explore more effective uses of this byproduct. Cajuput bark (*Melaleuca cajuputi* Powell) was sourced from a 26-year-old cajuput stand in Paliyan, Gunungkidul. The bark powder was extracted using a methanol-water solvent mixture at concentrations of 40% and 80%. Extraction was conducted in water baths at temperatures of 60°C and 100°C for 120 minutes. GC-MS identified several chemical components in the methanol extract of cajuput bark, including sugars (erythritol, D-arabinose, D-xylose, adonitol, d-galactose, D-glucitol, and L-rhamnose), fatty acids (lactic acid, glycerol, glyceric acid, malic acid, and palmitic acid), and phenolic derivatives (gallic acid and protocatechuic acid). The average extractive content of the methanol extract of cajuput bark was 4.07%, and the percentage inhibition of antioxidant activity was 40.26%.

Keywords: cajuput, cajuput bark, chemical compounds, GC-MS, extractive content, antioxidant activity

Introduction

Cajuput (*Melaleuca cajuputi* Powell) is a native Indonesian plant utilised primarily for cajuput essential oil production. This plant is also valuable for reforestation and agroforestry due to its economic importance and its ability to thrive in poor environmental conditions (Kartikawati *et al.* 2014). While the leaves, twigs, and branches are used in the essential oil distillation process, other parts of the plant, such as the fruits, flowers, bark, and roots, remain underutilised. In contrast, related species such as gelam bark (*Melaleuca cumingiana* Turcz.) have been studied for various applications, including activated carbon material (Abdullah 2001), antibacterial (Sudiansyah *et al.* 2023), natural dye (Nintasari and Purwanto 2016), and raw material for binderless bark particleboard (Christy *et al.* 2021).

All parts of the plant contain different chemical components. Analysing the chemical composition of the bark of various *Melaleuca* species is crucial to enhance its value as a raw material. Recent research by Zamzami *et al.* (2021) revealed that the methanol extract of gelam bark (*Melaleuca cumingiana* Turcz.) primarily consists of compounds such as polyphenols, flavonoids, alkaloids, tannins, steroids, and terpenoids. Similar findings are anticipated for *M. cajuputi* Powell.

The bark of many species is known to be composed of polyphenols, lignans, and antioxidants (Pietarinen *et al.* 2006). Antioxidants, in particular, are widely used as food additives to protect against oxidative degradation caused by free radicals and combat harmful reactive oxygen species in the human body (Luis *et al.* 2014). Solvent extraction is a common method for isolating various bioactive compounds,

including antioxidants, from plant bark. Solvent extraction is the simplest, easiest and fastest method to provide bioactive content with low cost. The most common solvents extraction methods used was methanol and ethanol as extractants on water bath. The biggest advantage of water bath are all factor can be accurately controlled and the reactant are heated evenly (Han *et al.*, 2020). In this study, extractive content and antioxidant activity of cajuput bark extract were investigated under different conditions. Chemical components composition was identified to explore potential methods for enhancing the utilisation of cajuput bark.

Materials and Methods

Sample preparation

Cajuput bark (*Melaleuca cajuputi* Powell.) was sourced from a 26-year-old cajuput stand, planted in 1998, in Paliyan, Gunungkidul. The bark was collected from the trunks of several trees after their leaves had been harvested for essential oil production. Using a machete, the bark was removed from the ground surface to a height of 1.3 m, revealing the inner bark. The bark was homogenised by mixing material from different trees and cutting it into small pieces. Finally, the cajuput bark was ground to a powder with a mesh size of 40–60 for the extraction process.

Extraction of cajuput bark

Two grams of cajuput bark powder were dissolved in a methanol-water solvent (40% and 80% v/v) in a 500 mL Erlenmeyer flask. Extraction was conducted in water baths for 120 minutes at 60°C and 100°C. After extraction, the

solution was filtered, and the filtrate was evaporated on a heating plate. The extractive content of cajuput bark was calculated by comparing the oven-dry weight of the extract with the dry weight of the initial powder.

Chemical Component Analysis

The chemical compounds in cajuput bark extract were analysed using GC-MS with the derivatisation method (Moldeveanu and David 2019). The derivatisation reagents consisted of trimethylchlorosilane (TMCS), N,O-Bis (trimethylsilyl) trifluoroacetamide (BSTFA), and pyridine. A 5 mg sample of cajuput bark methanol extract was diluted with 100 μ L of pyridine in an injection vial and sonicated. Then, 1 μ L of TMCS solution and 99 μ L of BSTFA solution were added to the vial, which was sonicated again. The vial was heated for 60 minutes at a temperature of $103 \pm 2^\circ\text{C}$. For GC-MS analysis, three different solvents were used: methanol, *n*-hexane, and acetone. The sample was injected into the GC-MS system with a RTX-5MS capillary column (30 m \times 0.25 mm \times 0.25 μ m; GL Sciences, Tokyo, Japan). The temperature was programmed to rise at a rate of 6°C per minute from 70°C to 290°C . The system was run with helium serving as the carrier gas, a separation ratio of 1:9.5, and a total retention time of 60.83 minutes. The analysis was carried out using the relative peak area method. Compounds were identified using the NIST 20 library (National Institute of Standards Technology).

Determination of DPPH inhibition antioxidant activity (IAA)

Antioxidant activity was determined using the DPPH (1 1-diphenyl-2-picrylhydrazyl) free radical scavenging assay (Gao *et al.* 2006). Cajuput bark extract was diluted in several concentrations (1000, 500, 250, and 125 ppm). Then, 3 mL of 4% (w/v) DPPH solution was mixed with 0.1 mL of each extract solution (A1). A₀ consisted of 0.1 mL of methanol and 3 mL of DPPH (blank absorbance). Methanol served as the absorbance blank. All samples were incubated in a dark at room temperature for 30 minutes. Absorbance was measured at 517 nm with a WPA 800+ spectrophotometer (A1). The test was carried out in duplicate. Catechin and tannic acid were used as positive controls for comparison. The percentage inhibition of antioxidant activity (%IAA) was calculated using the following equation:

$$\% \text{ Inhibition} = \frac{(A_0 - A_1)}{A_0} \times 100$$

Results and Discussion

Extractive content of cajuput bark

As shown in Table 1, the average extractive content (EC) of the cajuput bark methanol extract in this study was 4.07%, which was greater than the values reported by Arisandi *et al.* (2024), ranging from 1.00% to 1.03%, and by Batubara *et al.* (2012), which was 1.20%. In comparison, the average EC of the methanol extract from *E. globulus*, which belongs to the same family as cajuput, was reported to be 9.92% by Luis *et al.* (2014) and 2.7% by Vazquez *et al.* (2008).

Increasing the concentration of the methanol solvent from 40% to 80% at 60°C and 100°C resulted in increases in the extractive content from 1.65–2.15% to 5.45–6.77%, respectively. At the same methanol solvent concentration, the extraction temperature did not affect the EC value. This contradicted with Vergana-salinas *et al.* (2012) showed that the use of high temperature on extraction process did not damage the chemical compound but provided better extractive yield and antioxidant activity. Low methanol concentration (40%) at temperatures of 60°C and 100°C yielded EC values in the range of 1.65–2.06%, while high methanol concentration (80%) at the same temperatures increased the EC values to 5.45–6.77%. This suggests that the concentration of the methanol solvent had a considerable effect on the efficiency of the methanol extraction of cajuput bark. This finding confirmed the work of Arisandi *et al.* (2024), who observed that methanol solvents provided higher EC values for cajuput bark extraction than water solvents.

Several studies have reported higher EC values from bark extraction using methanol than other solvents, such as *n*-hexane, acetone, ethyl acetate, and ethanol (Vazquez *et al.* 2008; Luis *et al.* 2014; Arisandi *et al.* 2024). Additionally, the high EC value of the bark indicates that extractives in this part are more abundant than in the stem (Pereira *et al.* 2004). According to Lukmandaru (2011), extractives soluble in methanol solvents include highly polar compounds such as starch, sugars, and dissolved carbohydrates.

As explained by Piwowska and González-Alvarez (2012), increasing the solvent concentration enhances the solubility of polyhydrogen bonds, hydrophobic interactions with polysaccharides, and protein bonds in cell walls, facilitating the dissolution of extractive components. Consequently, higher amounts of dissolved substances lead to a higher EC value of the extracted sample.

Table 1. Extractive content and antioxidant activity of cajuput bark extract

Samples	Temperatures ($^\circ\text{C}$)	Duration (minutes)	Methanol Conc. (%)	Dry weight extract (g)	EC (%)	IAA at 1000 ppm (%)
A1(1)	60	120	40	0.04	1.65	35.13
A1(2)	60	120	40	0.05	2.15	26.41
A2(1)	60	120	80	0.13	5.77	45.04
A2(2)	60	120	80	0.15	6.77	43.90

B1(1)	100	120	40	0.04	2.06	43.60
B1(2)	100	120	40	0.04	2.06	44.50
B2(1)	100	120	80	0.15	6.64	41.00
B2(2)	100	120	80	0.12	5.45	42.52
Min				0.04	1.65	26.41
Max				0.15	6.77	45.04
Average				0.09	4.07	40.26
Standard deviation				0.05	2.23	7.04

Note: EC = extractive content; %IAA = percentage inhibition of antioxidant activity; bold = the highest value; italic = the lowest value.

Cajuput Bark Chemical Component Analysis

GC-MS analysis of the cajuput bark methanol extract was carried out using three different solvents. The injection with methanol as a solvent did not identify the chemical components in the cajuput bark sample and instead indicated

impurities as evidenced by a similarity index value of less than 40%. Consequently, the derivatisation method involving TMCS was employed, with *n*-hexane and acetone serving as solvents, to improve the results of the chemical composition identification.

Table 2. GC-MS result of Cajuput Bark Methanol Extract (TMCS Derivative)

No	Name	SI (%)	Formula	Concentration (%)	
				Acetone	Hexane
1	Lactic acid	91	C ₉ H ₂₂ O ₃	-	6.33
2	Boric acid	82	C ₉ H ₂₇ BO ₃	6.21	4.02
3	Glycerol	94	C ₁₂ H ₃₂ O ₃	15.06	19.01
4	Glyceric acid	92	C ₁₂ H ₃₀ O ₄	-	2.86
5	Erythritol	84	C ₁₆ H ₄₂ O ₄	3.23	2.42
6	D-Arabinose	85	C ₁₇ H ₄₂ O ₅	4.03	-
7	Malic acid	88	C ₂₂ H ₄₈ O ₅	3.07	-
8	D-Lyxose	84	C ₁₇ H ₄₂ O ₅	4.76	-
9	Adonitol	94	C ₂₀ H ₅₂ O ₅	7	6.33
10	Protocatechuic acid	85	C ₁₆ H ₃₀ O ₄	-	2.42
11	d-Galactose, 2,3,4,5,6-pentakis-O-(trimethylsilyl)-, o-methyloxyme, (1Z)-	91	C ₂₂ H ₅₅ NO ₆	8.76	5.51
12	D-Glucitol	91	C ₂₄ H ₆₂ O ₆	45.67	32.45
13	Gallic acid	92	C ₁₉ H ₃₈ O ₅	-	3.64
14	L-Rhamnose	85	C ₁₈ H ₄₄ O ₅	2.22	-
15	Palmitic acid	90	C ₁₉ H ₄₀ O ₂	-	15.01
Total (%)				100	100

Note: SI= Similarity Index (%)

Table 2 presents the results of the GC-MS analysis of the cajuput bark methanol extract using *n*-hexane and acetone as solvents. According to these results, cajuput bark consisted of two main groups of compounds: fatty acid and sugar groups. The analysis with both solvents identified glucitol (32.45–45.67%) and glycerol (15.06–19.01%) as the compounds with the highest concentrations in the cajuput bark extract. The *n*-hexane-soluble extract was identified to be richer in fatty acid compounds, while the acetone-soluble extract is richer in sugar compounds. Additionally, several simple phenolic compounds, such as protocatechuic acid and gallic acid, were detected only in the *n*-hexane-soluble extract.

Arisandi *et al.* (2024) studied the *n*-hexane extract of cajuput bark extracted by soxhlet extraction and identified

several lipophilic components, including hydrocarbons (13%), fatty acids (21.3%), fatty alcohols (9.63%), sterols and steroids (31.4%), triterpenoids (21.9%), and aldehyde compounds (1.7%). This study found similar results for fatty acid fractions. A clear difference was seen on the abundant of aldehyde, triterpenoid, sterols and steroids compound that appears when cajuput bark was extracted by *n*-hexane as extraction solvent. Other acid compounds were found as simple phenolic acids. Additionally, other studies have reported some phenolic derivatives in methanol extracts from plant bark. Yazaki and Hillis (1967) found that methanol extracts from the bark of several *Eucalyptus* species contained ellagic acid and gallic acid derivatives such as ellagitannin, methyl, and glycosyl. Similarly, methanol extracts from the bark of several plants in the *Myrtaceae*

family, such as *E. maidenii* and *E. urograndis*, contained quinic acid, gallic acid, protocatechuic acid, catechin, and ellagic acid (Santos *et al.* 2012). Those findings confirmed the presence of phenolic compounds and their derivatives in cajuput bark.

GC-MS analysis detected several sugar compounds in the cajuput bark methanol extract. The same sugar components were also identified in the bark of other species within the *Myrtaceae* family, to which cajuput belongs. Vazquez *et al.* (2008) reported that 60.96% of the *E. globulus* bark content was composed of sugars, including glucose, xylose, arabinose, galactose, and mannose. Miranda *et al.* (2016) similarly found that the bark extract of *E. sideroxylon* contained sugar groups, such as monosaccharides, glucose, xylose, galactose, arabinose, mannose, and rhamnose. The considerable amounts of sugar components identified in this study might be attributed to the use of polar solvents, such as methanol, which are effective in dissolving starch and sugars in plant extracts (Arisandi *et al.* 2024).

GC-MS analysis was able to identify only a few polar components of the cajuput bark methanol extract, suggesting that this method might not be fully effective for such identification. For more accurate identification, it is recommended to employ other tools, such as LC-MS. In addition, the presence of numerous impurities in the methanol extract solution might have contributed to this challenge, suggesting the need for pre-screening or purification of the methanol extract before injection into the GC-MS system.

Antioxidant activity of cajuput bark

The average percentage inhibition of antioxidant activity (%IAA) for the cajuput bark methanol extract at 1000 ppm in this study was 40.26%. Catechin compounds and tannic acid have standard percentage inhibition values at 1000 ppm of 85.65% and 90.30%, respectively, which are considerably higher than the value obtained in this study. This confirms that the cajuput bark methanol extract was weaker than the standards.

According to Table 1, increasing the methanol concentration from 40% to 80% at 60°C resulted in an increase in the %IAA value from 26.41–35.13% to 43.90–45.04%. However, at a higher temperature of 100°C, the %IAA value slightly declined from 43.60–44.50% to 41.00–42.52%. Meanwhile, at a methanol concentration of 40%, raising the temperature from 60°C to 100°C gave a boost to the %IAA value from 35.13–26.41% to 43.60–44.50%. On the other hand, at a methanol concentration of 80%, increasing the temperature in the same range caused the %IAA value to fall from 43.91–45.04% to 41.00–42.52%. These results show that both temperature and methanol concentration did affect the %IAA value. Specifically, at low solvent concentrations, an increase in extraction temperature increased the %IAA value. At high solvent concentrations, however, a rise in temperature lowered the %IAA value.

Several studies reported effective %IAA levels exceeding 70%. For instance, Hou *et al.* (2016) reported 86.0% IAA value for *M. braceteta* ethanol extract, which was higher than the IAA value observed in this study. Sulaiman *et al.* (2017) obtained an optimum IAA value of 72.95% for *Clinacanthus nutans* ethanol extract by using an ethanol solvent concentration of 10% for 120 minutes, whereas Yim *et al.* (2013) achieved a IAA value of 84.70% for *Schizophyllum commune* aqueous extract at a temperature of 35.7°C.

In this study, extraction was conducted within the temperature range of 60–100°C. The increase in antioxidant activity obtained at high temperatures most likely due to an increase in the total yield of the extract (Vergara-Salinas *et al.* 2012). As shown in Table 1 it appears that the samples with high extractive content provided better antioxidant value. Research has shown that effective antioxidant activity is generally reached in the extraction temperature range of 35–75°C with low solvent concentrations. It has been proven that the IAA value increased with the increasing temperature up to a certain point but decreased when the concentrations were high. Similarly, Sulaiman *et al.* (2017) reported that increasing the solvent concentration caused a slight decrease in the DPPH antioxidant activity. They also noted that pH and the chemical structure of DPPH also contributed to this decrease at high solvent concentrations. Therefore, the decrease in the IAA value in this study was likely caused by the interaction of high temperature and high solvent concentration.

The antioxidant activity can be affected by its chemical composition. The results of GC-MS identification showed that the antioxidant activity can be affected by the chemical composition of the extract. GC-MS analysis revealed that the cajuput bark methanol extract in this study was primarily composed of sugars and fatty acids, with small amounts of simple phenolic compounds such as gallic acid (3.64%) and protocatechuic acid (2.42%), which are known to exhibit antioxidant activity. Plants with high phenol and polyphenol contents typically exhibit strong antioxidant properties (Luis *et al.* 2014). These compounds possess redox molecular structures that act as reducing agents, hydrogen donors, and singlet oxygen quenchers, targeting free radicals. Therefore, the low antioxidant activity of the cajuput bark methanol extract observed in this study was likely due to the extract's low phenolic content. Furthermore, the presence of numerous inactive antioxidant compounds as well as active antioxidant compounds that were not fully dissolved in methanol as the extraction solvent might have contributed to low antioxidant activity. In addition, the method used might have influenced the results, suggesting the need for comparisons with other antioxidant activity test methods to have a better understanding of the antioxidant potential of cajuput bark.

Conclusion

Extraction of cajuput bark with methanol solvent using a simple extraction method revealed the presence of various chemical compounds, including sugars (erythritol, D-arabinose, D-lyxose, adonitol, d-galactose, D-glucitol, and L-rhamnose), fatty acids (lactic acid, glycerol, glyceric acid, malic acid, and palmitic acid), and phenolic derivatives (gallic acid and protocatechuic acid). The resulting methanol extract of cajuput bark had an extractive content of 6.77% and percentage inhibition of antioxidant activity of 45.04%. The low antioxidant activity value was likely due to the low phenolic content in the cajuput bark methanol extract, a high amount of impurities, and the presence of numerous inactive antioxidant compounds. Future research is suggested to quantify the phenolic compounds and their derivatives in cajuput bark extracts to better understand their antioxidant potential.

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Manuscripts will be accepted for publications are those discussing and containing results of research on wood science and technology, and reviews on specific topics, which are decided by the Editors and have not been published elsewhere. Authors are requested to correct the manuscripts accepted for publications as suggested by the Reviewers. Editors could change positions of Figures and Tables.

Manuscripts Preparations

1. Manuscripts must be in English, typewritten using Word, Arial Narrow, single space, 3 cm of left and right margin and 2.5 cm of top and bottom margin of a Letter paper size. Title is printed with a font size of 14 pt, Authors are of 12 pt, and Text is of 10 pt.
2. Manuscripts should be checked for spelling and grammar by a native speaker.
3. Manuscripts compositions:
 - 3.1. Title
 - 3.2. Complete name of Authors
 - 3.3. Abstract
 - 3.4. Key words
 - 3.5. Texts:
 - Introduction
 - Materials and Methods
 - Results and Discussion
 - Conclusions (and Suggestions)
 - References
 - Name and complete address of Authors
 - Appendix
 4. Other rules:
 - 4.1. Names of wood are followed by Botanical Name.
 - 4.2. Values between are written using this symbol (~), e.g. 3.75 ~ 8.92%.
 - 4.3. Editors could modify Figures without changing their substantial meaning.
 - 4.4. References are arranged from A to Z.
 - 4.5. References in text are written as this example: (Palomar *et al.* 1990; Arancon 1997).
 - 4.6. Examples of writing of References: Altschul, S.F.; T.L. Madden; A.A. Schäffer; J. Zhang; Z. Zhang; W. Miller; D.J. Lipman. 1997. Gapped BLAST and PSI-BLAST: A New Generation of Protein Database Search Programs. *Nucleic Acids Res.* 25: 3389-3402.

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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.

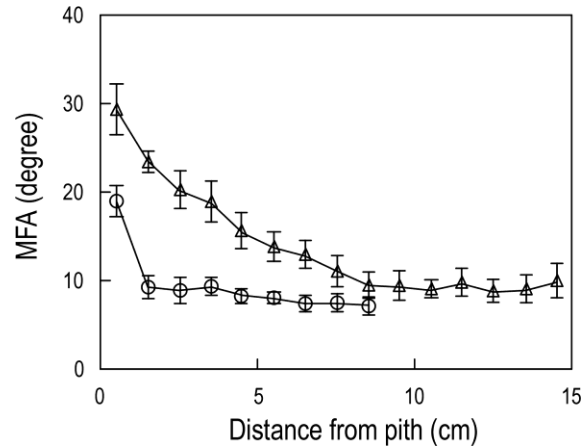


Figure 3. Radial variation of microfibril angle of the S2 layer in tracheid. Open circle, *Agathis* sp.; open triangle, *Pinus insularis*; Bars indicate the standard deviation. (Source: Ishiguri *et al.* 2010)

