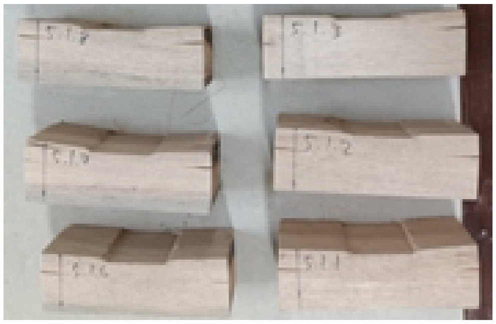
ISSN 2087-3840

WOOD RESEARCH Journal

Journal of Indonesian Wood Research Society

Volume 15, Number 1, April 2024



Volume 15, Number 1, April 2024 ISSN 2087-3840

WOOD RESEARCH Journal

Journal of Indonesian Wood Research Society

Chief Editor

Dr. Ratih Damayanti

Editorial Board Members

Prof. Yusuf Sudo Hadi
Prof. Bambang Subiyanto
Prof. Wasrin Syafii
Prof. Anita Firmanti
Prof. Nobuaki Hattori
Prof. Jugo Ilic
Prof. Edi Suhaimi Bakar
Prof. Sri Nugroho Marsoem
Prof. Imam Wahyudi
Dr. Farah Diba
Dr. Krisdianto
Dr. Ganis Lukmandaru
Dr. Tomy Listyanto
Fanny Hidayati, Ph.D.

Peer Reviewers

National Research and Innovation Agency
Prof. Subyakto
Prof. Myrtha Karina
Prof. Gustan Pari
Prof. Adi Santoso
Prof. Djarwanto
Prof. I.M. Sulastiningsih
Prof. Widya Fatriasari
Dr. Titik artika
Dr. Nanang Masruchin
Dr. Ina Winarni
Dr. Ismail Budiman
Dr. Novitri Hastuti
Dr. Gunawan Pasaribu
Dr. Jamaludin Malik
Dr. Dona Octavia
Dr. Maya Ismayanti
Dr. Didi Tarmadi
Dr. Muhammad Adly Rahandi Lubis
Dr. Kurnia Wiji Prasetiyo

IPB University
Prof. I Wayan Darmawan
Dr. Naresworo Nugroho
Dr. Istie S. Rahayu

Bengkulu University
Prof. Ridwan Yahya

Tanjungpura University
Prof. Yuliati Indrayani

Gadjah Mada University
Dr. Ragil Widyorini
Dr. M Navis Rofii
Dr. Hairi Cipta

Mulawarman University
Dr. Rudiantoro Amirta
Dr. Irawan W. Kusuma

Hasanuddin University
Prof. Musrizal Muin

Kyoto University
Prof. Junji Sugiyama
Prof. Toshiaki Umezawa
Prof. Takashi Watanabe
Dr. Misao Yokoyama

Dresden University
Dr. Christian Gottloober

Oklahoma State University
Prof. Salim Hiziroglu

Montpelier University
Prof. Joseph P. Grill

Duzce University
Prof. Cihat Tasciouglu, Ph.D

Kangwon National University
Prof. Nam-Hun Kim

University of Melbourne Prof.
Barbara Ozarska

Paris Tech Cluny
Prof. Remy Marchal

Universiti Putra Malaysia
Prof. Edi Suhaimi Bakar

Journal Manager	Dian Anggraini Indrawan, S.Hut., MM.
IT Manager	Suhardi Mardiansyah, A.Md.
Section Editors & Initial Reviewers	Fanny Hidayati, Ph.D. Dian Anggraini Indrawan, S.Hut., M.M.
Copy & Layout Editor	Suhardi Mardiansyah, A.Md.
Proof Readers	Prof. Subyakto Prof. Ganis Lukmandaru Dr. Krisdianto Dr. Tomy Listyanto
Treasurer	Dr. Firda Aulya Syamani, M.Si.

Study of the Effect of Grain Angle on the Compressive Strength of Red Meranti Timber (*Shorea spp.*)

Yosafat Aji Pranata, Anang Kristianto, Novi

Abstract

The compressive strength of timber is the main parameter in designing truss system, for instance timber bridges, building roof, or column in buildings. In term of design of compression structural components according to the SNI 7973:2013, the corrected compression design value is a calculation of compressive strength parameters and correction factors, for example, wet service factors, temperature factors, column stability factors, and others. Timber as an orthotropic material has three main directions, therefore the angle of the timber grain has an influence on compressive strength. This research aims to study the effect of timber grain's angle on the compressive strength of Red Meranti wood (*Shorea spp.*) and develop an empirical equation to calculate the compressive strength of timber with the influence of the wood grain's angle. The test specimens were made based on the primary method reference for compression test namely 50mm x 50mm x 200mm (parallel to the grain type), according to ASTM D143-22 for test specimens with variations in fiber direction, namely 0°, 10°, 20° and 30°. Meanwhile, test objects with variations in fiber direction, namely 60°, 70°, 80° and 90°, were made the sizes of 50mm x 50mm x 150mm (perpendicular to the grain type). Testings were carried out using a Universal Testing Machine with test speed according to ASTM D143-22. All test objects were made in dry conditions (moisture content ranging from 14% to 16%). The conclusion obtained from this research are an empirical equation for calculating the compressive strength of Red Meranti timber with a predictor is the timber grain's angle, which are $F_{CY} = 14.01 – 0.119\theta + 0.000042\theta^2$ (in term of yield of proportional point) and $F_{CU} = 29.82 – 0.417\theta + 0.0018\theta^2$ (in term of peak or ultimate point). This equation provides benefits for academics and practitioners, especially in designing compression structural components especially with compression value as the main parameter.

Keywords: Compression Strength, grain angle of timber, Red Meranti, Compressive Design, ASTM D143.

Introduction

Compressive strength of timber is the main parameter in designing of timber bridges (truss system), building roof (truss system), or column in buildings. In term of design of compression structural components according to the SNI 7973:2013 (BSN, 2013), the corrected compression design value is a calculation of compressive strength parameters and correction factors, for example, wet service factors, temperature factors, column stability factors, and others. Timber as an orthotropic material has three main directions, thus the angle of the timber grain has an influence on compressive strength.

Previous research related to timber properties, especially the compressive strength of Meranti species wood, has been carried out several times, namely experimental research on timber compression testing with several variations in grain angles (Pranata and Suryoatmono, 2012) for 3 (three) wood species, namely Acacia, Meranti, and Keruing. The results being an alternative of von Mises-based equation for calculating the compressive strength of wood, however this study was limited to only a few grain's angles and a limited number of test objects. Another study was experimental and numerical research to study the effect of grain's angle on the compressive strength of Red Meranti wood (Pranata and Suryoatmono, 2013) with variations in fiber angles of 12°, 60°, and 80°.

Wood is generally assumed to behave as mutually perpendicular material principal axes, namely and tangential axes. Compressive strength is the compressive force that acts on a unit cross-sectional area of wood that is subjected to that force. Compressive strength of wood defines the limit of wood's ability to accept compressive loads until the wood fails. Previous study of Red Meranti (*Shorea spp.*) compression strength were conducted by Nakai (Nakai, 1985), Chik (Chik, 1988), Pranata and Suryoatmono (Pranata and Suryoatmono, 2012; Pranata and Suryoatmono, 2013), Tjondro *et al.* (Tjondro *et al.*, 2016), Azmi *et al.* (Azmi *et al.*, 2022), and wood database (Meier, 2024). Table 1 shows the summary of compression strength of Red Meranti (*Shorea spp.*) timber obtained from previous research histories.

Table 1. Compression strength of Red Meranti (*Shorea spp.*) Timber from previous research histories.

References	θ (°)	F_{CY} (MPa)	F_{CU} (MPa)
Nakai, 1985	0	31.60	36.50
Azmi *et.al.*, 2022	0	31.30	-
Meier, 2024	0	33.90	-
Tjondro *et.al.*,	0	30.78	41.21
	90	7.51	-
Chik, 1988	0	-	39.60
	90	-	4.14

References	θ (°)	F_{CY} (MPa)	F_{CU} (MPa)
Pranata and Suryoatmono, 2012; Pranata and Suryoatmono, 2013	0	33.67	-
	5	31.16	-
	10	28.55	-
	12	27.82	33.30
	60	8.52	9.10
	80	7.68	8.10
	90	7.17	-

Currently, the parameters for compressive strength of timber are known parallel to the grain (grain's angle of 0°) or longitudinal direction, and compressive strength perpendicular to the grain (grain angle of 90°) or radial direction. These two parameters can be obtained from experimental testing in the laboratory using testing standards including ASTM D143-22 (ASTM, 2022) with primary and secondary test methods.

This research aims to study the effect of timber grain's angle on the compressive strength of Red Meranti wood (*Shorea spp.*) and develop an empirical equation to calculate the compressive strength of timber with the influence of wood grain's angle.

Materials and Methods

The scope of the research were that:

1. The timber studied is the Red Meranti species (*Shorea spp.*).
2. The total number of test objects are 45 test objects.
3. The test specimens were made based on the primary method reference for compression test specimens reference method 50mm x 50mm x 200mm (parallel to the grain type), according to ASTM D143-22 regulations for test specimens with variations in fiber direction, namely 0°, 10°, 20° and 30°. Meanwhile, test objects with variations in fiber direction, namely 60°, 70°, 80° and 90°, were made with test object sizes of 50mm x 50mm x 150mm (perpendicular to the grain type).
4. Testings are carried out using a Universal Testing Machine with test speed according to ASTM D143-22.
5. All test objects were made in dry conditions (moisture content ranging from 14% to 16%).
6. The compressive strength referred to in this research is the compressive stress calculated under peak or ultimate load condition (F_{CU}) and proportional load condition (F_{CY}).
7. Test objects are made from timber log, with angle dimensions adjusted for testing purposes.

Compression Tests

Testing was 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 compressive axial load and axial deformation. Figure 1 shows the test equipment used in this research. Figure 2a shows a schematic history of the load vs deformation relationship curve obtained from experimental test results.

Next, the curve was then converted into a curve for the relationship between stress and strain, where stress (engineering stress) is the compressive axial force divided by the initial cross-sectional area, while strain is the change in length (in this case shortening) divided by the initial length of the test object. Figure 2b shows a schematic of the stress vs strain relationship curve resulting from the conversion of the load vs deformation relationship curve.

Stress and strain were calculated using Equation 1 and Equation 2 (Goodno and Gere, 2021).

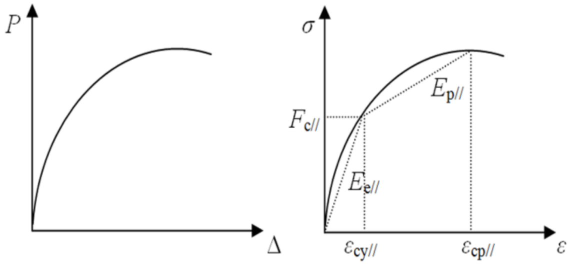
$$\sigma = P / A \qquad (1)$$

$$\varepsilon = \Delta / L_o \qquad (2)$$

with σ is engineering stress (MPa), P is axial compressive load (N), A is specimen's cross-section (mm^2), ε is strain (mm/mm), Δ is the change in length of shortening (mm), and L_o is initial length of the specimen (mm).

The testing speed (according to the ASTM D143's primary method) for the test object type parallel to the grain was a strain rate of 0.003 mm/mm per minute or a displacement rate of 0.6 mm per minute. While for the test object type perpendicular to the grain, the speed was a displacement rate of 0.305 mm per minute (ASTM, 2022).

Figure 1. Instrumen for testing using Universal Testing Machine (UTM)



(a). Load-Deformation Curve (b). Stress-Strain Curve

Figure 2. Idealization of the axial load vs axial deformation curve and normal (axial) stress vs strain (Pranata and Suryoatmono, 2013).

Proportional Load and Ultimate Load

The proportional point indicates when material behavior changes from elastic to plastic. One of the methods to calculate the proportional point is the Yasumura and Kawai Method (Munoz *et al*., 2010). The calculated initial stiffness was between 10% and 40% of the ultimate or peak load. A straight line between 40% and 90% of the peak load and a straight-line tangent to the load-displacement curve, then parallel to the 40% and 90% second line, were determined.

In this research, this method was used to determine the proportional load divided by a cross-section's area, to calculate the compression strength in terms of yield or proportional strength. While the compression strength in terms of ultimate strength was calculated using peak load, divided by cross-section's area..

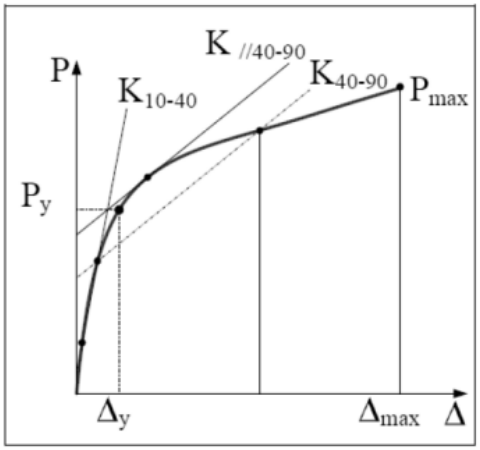


Figure 3. Yasumura and Kawai Method to determine the proportional load of timber (Munoz *et.al*., 2010).

Polynomial Regression Analysis

Several parameters for statistical data, which are mean, standard deviation, and coefficient of variation, are needed for analysis. Standard deviation measures how they are distributed around the arithmetic mean (Heumann et.al., 2017). A low standard deviation value indicates that the values are highly concentrated around the mean. Meanwhile, the coefficient of variation (usually expressed as a percentage) is a ratio between the standard deviation and the average value. In this research, polynomial regression analysis was carried out with Minitab software (LLC, 2023).

Results and Discussion

The test specimens with an angle of less than 45° were made and tested based on the primary method reference for compression test specimens reference method parallel to the grain type according to ASTM D143-22 regulations. This method used for test specimens with variations in fiber direction, namely 0°, 10°, 20° and 30°.

Meanwhile, test objects with an angle of more than 45° with variations in fiber direction, namely 60°, 70°, 80° and 90°, were made and tested with perpendicular to the grain type in accordance with ASTM D143-22. All test objects were made in dry conditions (moisture content ranging from 14% to 16%). Test objects are made from timber log, with angle dimensions adjusted for testing purposes.

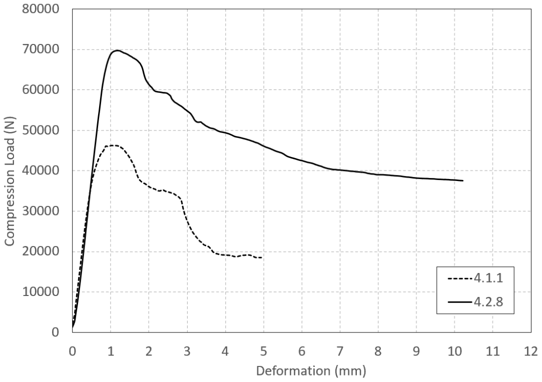


Figure 4. Tests results: Axial load vs deformation curve, obtained from experimental test for specimen 4.1.1 (grain's angle of 30°) and specimen 4.2.8 (grain's angle of 10°)

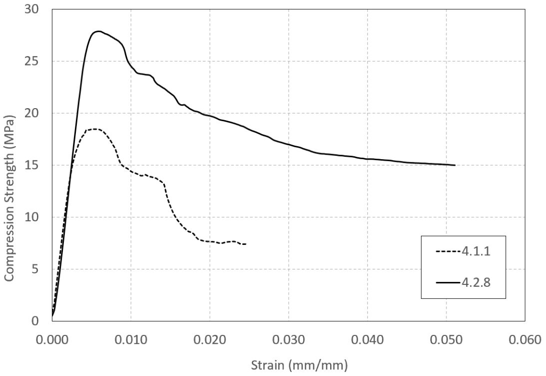


Figure 5. Convertion results: Stress vs strain curves of specimen 4.1.1 (grain's angle of 30°) and specimen 4.2.8 (grain's angle of 10°)

Figure 4 shows an example result obtained from compression test which is axial load vs deformation curve. Figure 5 shows a conversion results, which is calculation of stress and strain curves. The results above show that the test object with a lower grain angle produces a higher peak load than the test object with a larger grain angle.

Figure 6 shows an example of compression test using parallel to the grain method, while Figure 7 shows an example of compression test speciment with grain angle of 70°. Figure 8 shows some of test results of the specimens with dimension 50mm x 50mm x 200mm, while Figure 9 shows some of test results of the specimens with dimension 50mm x 50mm x 200mm.

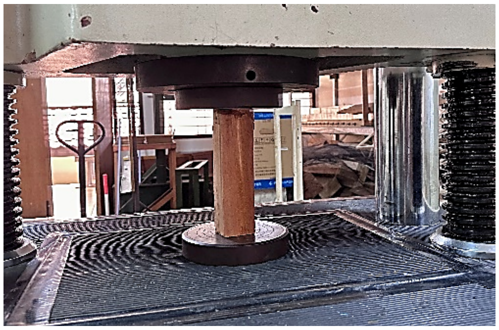


Figure 6. Experimental tests for 50 x 50 x 200mm specimen, using test method of compression parallel to the grain

Figure 7. Example of 70° grain angle of Red Meranti timber specimen

Figure 8. Results of failure mode for some 50 x 50 x 200mm specimens (compression parallel to the grain method of test)

Figure 9. Results of failure mode for some 50 x 50 x 150mm specimens (compression perpendicular to the grain method of test)

In this research, the compression strength were calculated in term of proportional load. The method to determine the yield point were cariied out by using Yasumura and Kawai method (Munoz et.al., 2010), and in term of peak of ultimate load. Table 1 shows the results of calculation of the proportional load, peak load, deformation at proportional load, deformation at peak load, and the grain's angle for all 45 specimens. Table 2 shows the results of calculation of the stress and strain in term of proportional stress and peak stress using Equation 1 and Equation 2.

Table 2. Tests results: Load and deformation obtained from experimental tests and calculation using Yasumura and Kawai Method

Specimens	P_y (N)	P_{peak} (N)	D_y (mm)	D_{peak} (mm)	θ (°)
1	14313.0	26650.6	0.3	5.0	70
2	7932.0	18839.9	0.7	6.0	80
3	18834.1	32409.2	0.4	6.2	60
4	13788.4	25203.8	1.5	6.0	70
5	15479.0	25547.9	0.5	6.0	70
6	29603.0	46987.6	0.3	0.8	30
7	28950.5	46224.6	0.4	1.0	30
8	24203.2	43516.9	0.3	0.8	30
9	123246.1	69692.6	1.6	1.2	10
10	5906.3	14661.3	0.7	6.0	90
11	17531.7	39520.9	0.8	6.1	30
12	16969.8	32393.3	0.5	6.0	60
13	10581.1	21456.7	0.5	6.0	80
14	6994.9	13948.3	1.5	6.0	90
15	37925.6	62397.2	0.4	0.8	10
16	38100.2	53651.3	0.5	0.9	20
17	32993.8	53975.8	0.3	0.8	20
18	56268.3	62644.0	0.7	1.1	10
19	16092.7	33444.2	0.4	6.1	60
20	6041.2	14045.7	1.3	6.1	90
21	5608.1	13160.7	1.2	6.1	90
22	18349.4	27524.6	1.2	6.1	70
23	36903.4	73806.8	0.3	0.8	0
24	39992.2	79984.5	0.8	1.7	0
25	29505.0	77361.8	0.8	1.6	0
26	26908.8	70598.3	0.4	1.1	10
27	28731.6	78258.5	0.5	1.3	0
28	29826.7	80297.5	0.5	1.3	0
29	21269.1	48193.7	0.4	6.1	30
30	24211.4	53836.4	0.5	6.1	20
31	44493.6	74663.0	0.4	0.7	0
32	32710.6	65421.2	0.5	14.5	10
33	40336.6	52428.3	0.4	0.9	20
34	35617.8	71235.7	5.3	1.1	10
35	12995.6	32007.6	0.5	6.1	60
36	6229.3	11832.2	1.4	6.1	90
37	15569.4	27696.7	0.5	6.1	70
38	13269.9	25523.0	0.4	6.2	70
39	11873.1	21078.7	0.7	6.1	80
40	9391.3	17105.2	0.5	6.1	90
41	11103.5	19313.0	0.4	6.1	80

Spec-imens	P_y (N)	P_{peak} (N)	D_y (mm)	D_{peak} (mm)	θ (°)
42	18469.9	44526.6	0.6	2.2	30
43	79513.1	46903.8	1.6	1.2	20
44	15292.9	30585.8	0.3	0.9	60
45	19199.5	31501.1	0.3	1.0	60

Table 3. Conversion results: Stress and Strain at proportional and ultimate limit conditions

Spec-imens	F_{CY} (MPa)	F_{CU} (MPa)	ε_y (mm/mm)	ε_U (mm/mm)	θ (°)
1	5.6	10.4	0.002	0.025	70
2	3.1	7.4	0.003	0.030	80
3	7.4	12.7	0.002	0.031	60
4	5.4	9.9	0.008	0.030	70
5	6.1	10.1	0.002	0.030	70
6	11.6	18.4	0.002	0.005	30
7	11.3	18.0	0.002	0.007	30
8	9.6	17.2	0.002	0.006	30
9	13.9	27.8	0.010	0.008	10
10	2.3	5.8	0.004	0.030	90
11	7.0	15.7	0.004	0.031	30
12	6.8	12.9	0.003	0.030	60
13	4.2	8.4	0.003	0.030	80
14	2.7	5.5	0.007	0.030	90
15	14.8	24.4	0.003	0.005	10
16	15.2	21.4	0.003	0.006	20
17	13.2	21.7	0.002	0.006	20
18	12.1	24.6	0.005	0.008	10
19	6.6	13.7	0.002	0.030	60
20	2.5	5.7	0.007	0.030	90
21	2.2	5.2	0.006	0.031	90
22	7.2	10.8	0.006	0.030	70
23	15.0	29.9	0.002	0.005	0
24	15.7	31.4	0.005	0.012	0
25	11.7	30.6	0.005	0.011	0
26	10.6	27.9	0.003	0.007	10
27	11.5	31.3	0.003	0.009	0
28	12.0	32.4	0.003	0.009	0
29	8.8	19.2	0.002	0.030	30
30	9.4	20.9	0.002	0.030	20
31	17.4	29.1	0.002	0.005	0
32	12.8	25.5	0.003	0.096	10
33	15.4	20.1	0.002	0.006	20
34	13.6	27.2	0.035	0.008	10
35	5.3	13.1	0.002	0.030	60
36	2.6	4.9	0.007	0.030	90
37	6.4	11.4	0.002	0.031	70
38	5.4	10.4	0.002	0.031	70
39	4.9	8.6	0.003	0.030	80
40	3.9	7.0	0.002	0.030	90
41	4.5	7.9	0.002	0.031	80
42	7.6	18.2	0.004	0.015	30
43	9.6	19.2	0.011	0.008	20
44	6.3	12.6	0.002	0.006	60
45	7.8	12.8	0.002	0.007	60

Figure 10 shows the result obtained experimentally (F_{CY}) and the equation-curve obtained from the polynomial regression analysis to predict the value of compression strength of Red Meranti timber in term of proportional or yield point, this empirical equation result shows the relationship between the compression strength (unit in MPa) and the grain angle θ (unit in degrees). The coefficient of R^2 is generally it is relatively near 100%, for timber this is considered normal because timber is a material that comes from nature. The regression equation for the curve in Figure 10 is shown in Equation 3 and Equation 4.

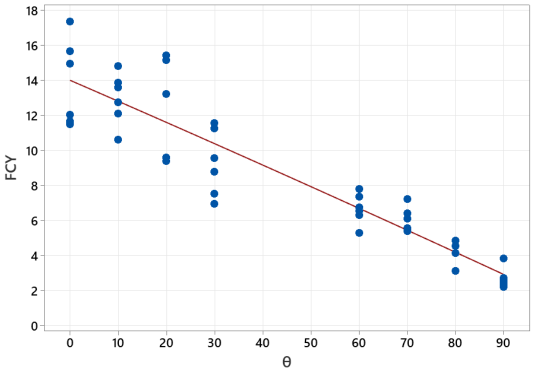


Figure 10. Results obtained from polynomial regression analysis.

$$F_{CY} = 14.01 - 0.119\theta + 0.000042\theta^2 \quad (3)$$

$$R^2 = 85.52\% \quad (4)$$

Figure 11 shows the result obtained experimentally (F_{CU}) and the equation-curve obtained from the polynomial regression analysis to predict the value of compression strength of Red Meranti timber in term of peak or ultimate point, this empirical equation result shows the relationship between the compression strength (unit in MPa) and the grain angle θ (unit in degrees). The coefficient of R^2 is generally it is relatively near 100%, for timber this is considered normal because timber is a material that comes from nature. The regression equation for the curve in Figure 10 is shown in Equation 5 and Equation 6.

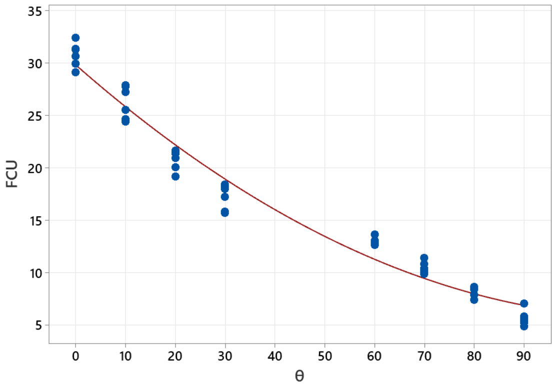


Figure 11. Results obtained from polynomial regression analysis.

$$F_{CU} = 29.82 - 0.417\theta + 0.0018\theta^2 \quad (5)$$

$$R^2 = 96.56\% \quad (6)$$

Conclusions

The conclusion obtained from this research is an empirical equation for calculating the compressive strength of Red Meranti timber (*Shorea spp.*) with a predictor, namely the wood grain's angle, namely $F_{CY} = 14.01 - 0.119\theta + 0.000042\theta^2$ with $R^2 = 85.52\%$ in term of yield or proportional point. While in term of peak or ultimate load, an empirical equation is $F_{CU} = 29.82 - 0.417\theta + 0.0018\theta^2$ with $R^2 = 96.56\%$.

F_{CY} or compression strength in term of proportional load value is an useful parameter for design of column or compression member in timber building, timber bridge truss, or timber roof truss in accordance with SNI 7973:2013. This equation provides benefits for academics and practitioners, especially in designing compression structural components, which is the compression design value parameter.

Acknowledgements

The authors received an internal funding research grant from Universitas Kristen Maranatha in the 2022 fiscal year, which was B-Scheme Research with the title "Experimental Study of the Compressive Mechanical Properties of Timber with Various Angle Direction". For this reason, largely author would like to express his deepest gratitude, so that all research activities can be completed in the same year. The authors also would like to thank all parties who have helped with the research process in the laboratory.

References

American Standard Testing and Material. 2022. ASTM D143-22 Standard Test Methods for Small Clear Specimens of Timber, West Conshohocken, Pennsylvania, United States, 2022.

American Standard Testing and Material. 2018. ASTM D5764-97a Standard Test Method for Evaluating Dowel-Bearing Strength of Wood and Wood-Based Products, West Conshohocken, Pennsylvania, United States, 2018.

Azmi, A., Ahmad, Z., Lum, W.C., Baharin, A., Za'ba N.I.L., Bhkari, N.M., Lee, S.H. 2022. Compressive strength characteristic values of nie structural sized Malaysian tropical hardwoods, Forests 2022, 13(8), 1172; https://doi.org/10.3390/f13081172.

Bodig, J., Jayne, B.A. 1993. Mechanics of Wood and Wood Composites, Krieger Publishing Company, Malabar, Florida, USA, 1993.

Chik, E.A.R. 1988. Basic and grade stresses for some Malaysian timbers. Malayan Forest Service Trade Leaflet No.38. The Malaysian Timber Industry Board And Forest Research Institute Malaysia, Kuala Lumpur 13 pp.

Goodno, B.J., Gere, J.M. 2021. Mechanics of Materials 9th Edition, ISBN-13: 9780357377857, Cengage Learning Asia.

Heumann, Christian, Michael Schomaker, Shalabh. 2017. Introduction to Statistics and Data Analysis: With Exercises, Solutions and Applications in R. Introduction to Statistics and Data Analysis: With Exercises, Solutions and Applications in R. Springer International Publishing, 2017.

LLC. 2023. Product version Minitab® 21.4.1 (64-bit) 2023 Minitab, LLC. All rights reserved.

Meier, E. The Wood database, URL: https://www.wood-database.com/worlds-strongest-woods, accessed on 15 October 2024.

Munoz, W., Mohammad, M., Salenikovich, A. & Quenneville, P. 2010. Determination of Yield Point and Ductility of Timber Assemblies: In Search for a Harmonized Approach, Engineered Wood Products Association, 2010.

Nakai, T. 1985. Mechanical properties of tropical woods. JARQ Volume 18 No. 4, pp. 315-323, 1985.

Pranata, Y.A., Suryoatmono, B. 2013. Nonlinear finite elemen modeling of red meranti compression at an angle to the grain. Journal of Engineering and Technological Science, Volume 45 No. 3, pp. 222-240, 2013.

Pranata, Y.A., Suryoatmono, B. 2012. distortion energy criterion for timber uniaxial compression mechanical properties. Jurnal Dinamika Teknik Sipil, Volume 12 No. 02, May 2012 (in Indonesian).

Tjondro, A., Suryoatmono, B., Imran, I. 2016, Non-linier Compression Stress-Strain Curve Model for Hardwood, Jurnal Teknik Sipil Mekanika, Volume 1 No. 1, pp. 49-52, June 2016.

Yosafat Aji Pranata
Universitas Kristen Maranatha
Jl. Suria Sumantri 65, Bandung, 40164, Jawa Barat

Tel.   : +62-22-2012186 ext. 1212
E-mail : yosafat.ap@gmail.com

Anang Kristianto
Universitas Kristen Maranatha
Jl. Suria Sumantri 65, Bandung, 40164, Jawa Barat
Tel.   : +62-22-2012186 ext. 1212

Novi
Universitas Kristen Maranatha
Jl. Suria Sumantri 65, Bandung, 40164, Jawa Barat
Tel.   : +62-22-2012186 ext. 1212

Chemical Properties of “Jati Unggul Nusantara” Teak Wood from Gunungkidul

Ganis Lukmandaru, Ardhan Wira Pratama, JP Gentur Sutapa, Sigit Sunarta, Denny Irawati, Rini Pujiarti, & Rizki Arisandi

Abstract

Jati Unggul Nusantara (JUN) trees show a fast-growing characteristic and a possibility to be harvested in short rotation. This study aims to determine the chemical properties of 8-year-old JUN tree parts. Three individual trees were felled from Paliyan, Gunungkidul, Yogyakarta, Indonesia. The tree parts were observed vertically (i.e., the bottom, center, top, branches, and twigs) and radially (i.e., sapwood and heartwood of the trunk). The result showed that the average content of cell wall components of extractive-free wood, i.e., hemicellulose, α-cellulose, and lignin, were 20.38~25.71%, 41.88~49.10%, and 26.46~29.85%, respectively. Furthermore, successive extractive measurements showed that ethanol-toluene and hot-water soluble extracts (based on dry wood) were at the levels of 3.01~7.58% and 1.85~3.09%, respectively. The ash content, silica content, and pH values were 0.48~0.82%, 0.13~0.37%, and 5.89~7.51%, respectively. By an analysis of variance, significant differences between the sapwood and the heartwood were observed in ethanol-toluene extractive, lignin, holocellulose, and cellulose contents. The differences among tree parts did not show any significant effect on the hot-water soluble content. Significant differences between the main stem and branches or between the main stem and twigs were observed in most chemical properties.

Keywords: Ash, cell wall component, extractive, fast-growing, juvenile

Introduction

Teak (*Tectona grandis* L.f.) wood is known as a fancy wood due to its wood strength, physical appearance, and high natural durability. The increasing demand for teak timber has caused an imbalance of supply and demand in the wood market (Thulasidas and Bailleres 2017). Consequently, the price of teak wood from long-rotation harvesting is increasing. Therefore, teak stands with fast-growing or superior trees have been developed by breeding to produce short-rotation teak trees.

Jati Unggul Nusantara (JUN) trees have been cultivated by shoot cuttings from Jati Plus Perhutani (JPP) clones (Efansyah *et al*. 2012). JUN trees have various advantages, such as faster growth with harvest periods of less than 10 years and deep-rooted physical characteristics (Anonymous 2011). Although it is harvested at a young age, the heartwood appearance indicates that the teak wood is considered to be durable enough to be used as the main raw material for construction and furniture.

In the logging practice, the main stems of young teak trees are mostly used for construction, while the branches and twigs are generally used as firewood. Therefore, it is important to assess the chemical properties of the wood as it can affect the woodworking process, colour, and natural durability. Studies on the chemical components of JUN and other fast-growing teak trees in general are still limited when compared to studies on their physical properties. Research using 5-year-old JUN trees from a coppiced stand was carried out for chemical properties of their wood by Maulida *et al.* (2020). Recently, the extractive contents of JUN wood (8 years) have been studied by Rahman *et al*. (2022). As a follow-up, this study aims to determine the chemical properties of the main stem, branches, and twigs of JUN trees. Better utilization of JUN wood is expected after evaluation of the chemical properties.

Materials and Methods

Samples Preparation

The materials used in this study were three individual JUN trees (8 years) from RPH Kepek, BDH Playen, BKPH Yogyakarta. Wood blocks of the main trunk (dbh = 14~18 cm, heartwood proportion = 33~49%) were cut from the bottom (1 m above the ground), center (2 m from the bottom), and top (2 m from the center) parts. The blocks from branches and twigs were taken from near the top part (Fig. 1). The wood from the main stem was divided into heartwood and sapwood. After removing the bark, each part of the tree was then converted into wood powder (40~60 mesh) by grinding for chemical evaluation.

Cell Wall Components and Extractives Determination

The determination of extractive contents was carried out by extracting wood powder (2 g equiv. dry weight) with ethanol-toluene solvent (2:1, v/v) (ASTM D1107 – 96 2002) and with hot water (ASTM D 1110 – 80 2002) in succession. The extractive-free wood powder from ethanol-toluene extraction was then determined for holocellulose content using the modified chlorite acid method (Browning 1967), as well as for α-cellulose (Rowell *et al.* 2005) and Klason lignin contents (TAPPI T222 – os 78 1992). The hemicellulose content was determined by subtracting the holocellulose content by the α −cellulose content. The ash content (ASTM D 1102 – 84 2002) as well as the silica content (SNI 14-1031-1989, Dewan Standarisasi Nasional 1989) were measured.

pH Value Measurement

The pH value was measured with the OAKTON pH tester. A total of 1 g (equiv. oven-dry wood) was soaked in 20 ml of distilled water for 48 hours. The pH value of the filtrate was then measured after filtering.

Data Analysis

A one-way analysis of variance (one-way ANOVA) was carried out to determine the effect of tree part factor i.e. bottom-heartwood, bottom-sapwood, center-heartwood, center-sapwood, top-sapwood, top-heartwood, branch, and twig parts. The effects were taken into account when significant at the 95% level (Type III Sums of Squares). A Tukey test (honestly significant difference) was used to show which group means differ. All calculations used SPSS 16 for Windows software.

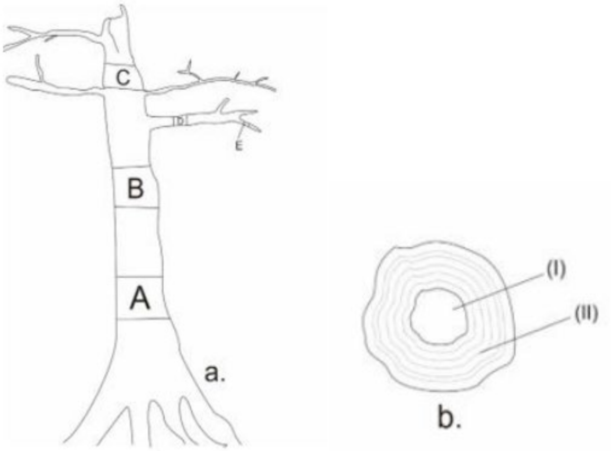


Figure 1. Sampling position on a (a) tree part (A=bottom, B=center, C=top) and cross-section (b) of teak trunk (I=heartwood, II= sapwood)

Results and Discussion

Chemical properties from fast-growing teak trees

The 8-year-old JUN wood was assumed to be juvenile wood (Bhat 20001), which was selected to show different properties than those of mature wood (Shmulsky and Jones 2011). A summary of the chemical properties of the 8-year-old JUN wood in this experiment and a comparison with those of fast-growing teak from previous works are presented in Table 1.

The JUN samples from Gunungkidul had higher levels of extractives, holocellulose, and α-cellulose compared to the 5-year-old JUN main stem samples from a coppiced stand (Maulida *et al.* 2020). They also had higher extractive content but lower lignin content compared to the 10-year-old samples from a community forest (Rizanti *et al.,* 2018). Furthermore, they had lower amounts of soluble ethanol-toluene extractives, holocellulose, hemicellulose, ash, and silica compared to 15-year-old Jati Plus Perhutani (superior teak) wood samples.

In the branch part, they showed holocellulose, α-cellulose, and lignin contents that were within the range comparable to those of branches of the api wood or *Schizolobium amazonicum* species (Amin *et al.* 2013). Research was also carried out on pohon surga (*Ailanthus altissima)* wood, where the branches and twigs had ash contents of 1.75% and 1.25%, respectively (Samariha and Kiaei2011).

Wood quality is affected by its extractive content. The higher the extractive content, especially the ethanol-toluene extract, the higher the durability or resistance of the wood to pathogens. Extractives in cell walls are also able to increase density and reduce swelling and shrinkage of wood (Shmulsky and Jones 2011). The low value of the ethanol-toluene extractive content of the 8-year-old JUN wood in this study is thought to reduce the natural durability of the wood when compared to the natural durability of mature teak. However, the relatively low value of hot-water extractive content in the JUN wood is assumed to provide benefits when used in wood gluing products. Previous studies demonstrated that non polar extractives cause change in polarity and wettability to result poor adhesion (Hse and Kuo 1988).

Table 1. Chemical properties of teak wood (Jati Unggul Niusantara) with references of teak wood chemical properties from fast-growing trees.

References			Jati Unggul Nusantara (8 years)			Content (%)
15 years[3]	10 years[2]	5 years[1]	Twig	Branch	Wood	
7.91~9.74	1.6(0.05)[5]	1.2(0.06)[4]	5.85	3.53	3.01~7.58	Ethanol-toluene Extractive [b]
2.02~2.42	2.5(0.48)	2.5(0.48)	2.08	1.85	2.16~2.74	Hot-water solubility[b]
75.93~77.47	67.50(1.53)	63.11~64.08	66.70	63.12	66.49~71.27	Holocellulose[a]
47.07~47.64	48.80(1.19)	33.68~41.16	41.88	47.74	43.57~49.10	α-cellulose[a]
26.87~29.86	18.70 (1.72)	-	24.48	20.38	21.62~25.71	Hemicellulose
28.12~32.45	35.53(0.78)	24.33~30.54	27.36	27.56	26.46~29.85	Lignin[a]
0.86-1.18	-	-	0.77	0.80	0.48~0.82	Ash[b]
0.36~0.55	-	-	0.22	0.32	0.18~0.37	Silica[b]
7.08~7.38	-	-	5.89	5.92	6.9~7.51	pH value

Remarks: Average of three replications (with the standard deviation parentheses) a = percentage of free-extractive meal; b = percentage of oven-dry weight meal

Sources: [1]Maulida *et al.,* (2020): coppiced superior teak (Jati Unggul Nusantara),
[2]Rizanti *et al*. (2018): from fast-growing clonal seeds grown in a community forest;
[3]Lukmandaru *et al.* 2016: Jati Plus Perhutani (superior teak) from Perhutani plantation
[4]ethanol-benzene (1/2,v/v) extraction,
[5]successive extraction with dichloromethane, acetone, and ethanol/toluene(1/2, v/v)

The pH values observed here were relatively high and close to neutral pH, while the ash and silica content values were relatively low. In an earlier study, pH value range of teak wood from community forests in Gunungkidul were 5.23~6.98 (Lukmandaru *et al*. 2017). It is thought that the site influenced the pH values. High levels of ash content can be the main inhibitor in the hardening process of urea resin adhesive in teak wood (Kanazawa *et al.* 1978). The low ash content of the JUN wood in this study is an advantage in wood bonding. Similar to the ash content, lower levels of silica content would reduce the negative effect on sawmill equipment (Shmulsky and Jones 2011) because most samples showed silica levels below 0.3% and only one sample showed values above 0.3% (the bottom of the sapwood part, 0.37%). Technically, a low pH value will accelerate the hardening of UF adhesive in the wood bonding process during hot pressing for particleboard (Maloney 1993). In contrast to wood-cement compatibility, a lower pH value will tend to inhibit the crystallization process compared to a high pH value (Hachmi and Moslemi 1990). In this study, the JUN wood had a relatively high percentage of sapwood (51~78%) and a pH value in the range of 6.90~7.51. It is thought that this will tend to reduce the speed of the UF adhesive hardening process. Likewise, low pH values tend to reduce the hardening process of UF adhesive on particleboard. However, low pH values will benefit the compatibility of wood with cement.

Comparison of Tree Parts

The results of the one-way ANOVA on the main stem parts showed a significant effect on all parameters except for solubility in hot-water. Post-hoc Tukey test results are shown in Figure 2~4. Based on the Tukey test results of the main stem parts for the vertical position, significant differences existed in all properties except for pH values. The amounts of ethanol-toluene extractive, holocellulose, hemicellulose, and lignin contents decreased from the bottom to the top in the heartwood parts. A similar pattern was observed in ash and silica contents for the sapwood parts. The cellulose content fluctuated both in sapwood and heartwood. A slightly different pattern was observed in the 5-year-old JUN samples, in which case the levels of holocellulose and lignin tended to be high in the center (Maulida *et al.* 2020) (Table 1). Syahidah *et al.* (2007) observed teak wood < 10 cm in diameter from community forests in South Sulawesi and found the highest cellulose content at the top part. The high content of cellulose and lignin in the middle heartwood might be due to variation on specific gravity and juvenility (Ona *et al.* 1998; Rahman *et al.* 2018). Similar pattern was also observed in raru wood (Iswanto *et al.* 2021).

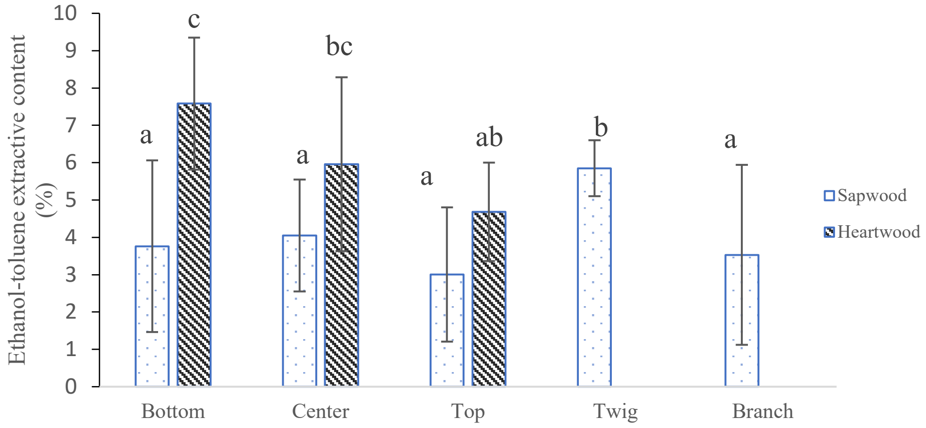


Figure 2. Ethanol-toluene extractive content (% oven dried wood) of Jati Unggul Nusantara (Superior teak) wood by tree part. Average of 3 trees, with the standard deviation error bar. The same letters on the same graphic are not statistically different at $p < 0.05$ by Tukey's test.

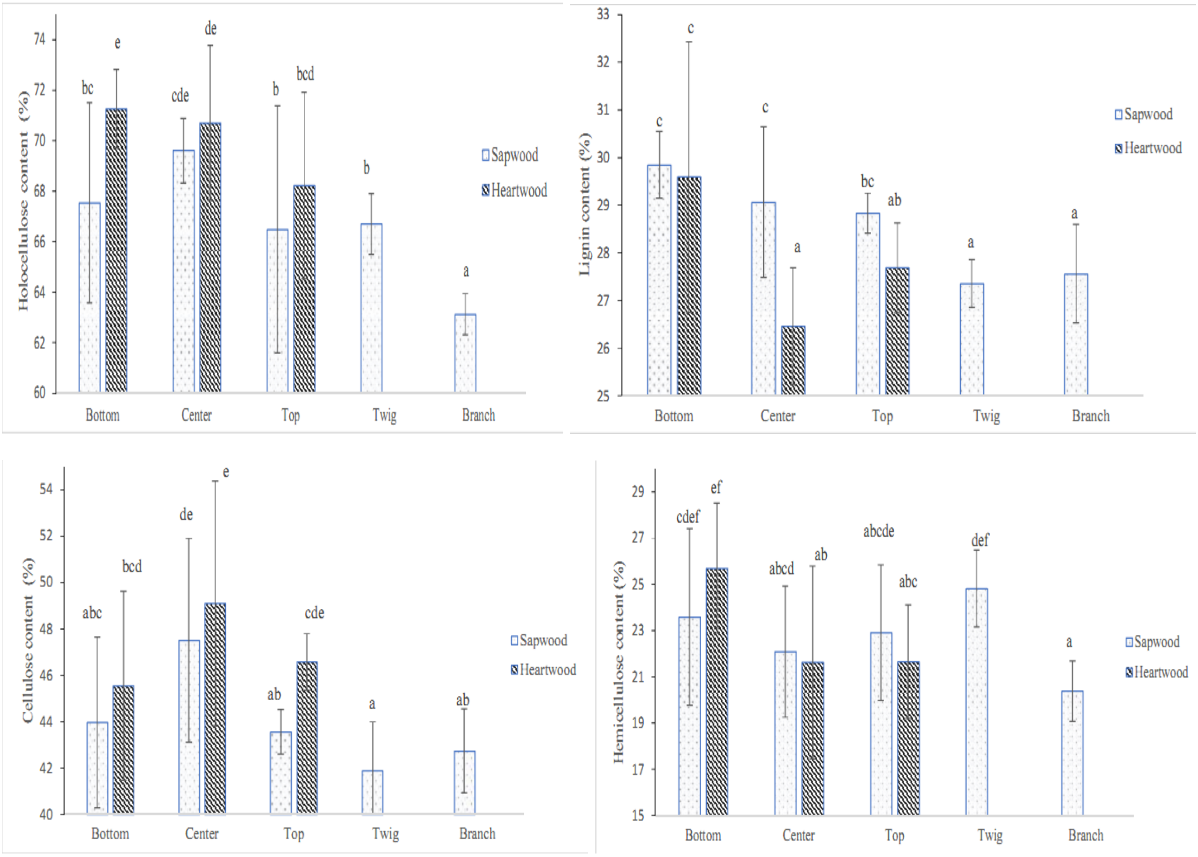


Figure 3. Cell wall component contents (%, extractive-free wood) of Jati Unggul Nusantara (Superior teak) wood by tree part. Average of 3 trees, with the standard deviation error bar. The same letters are not statistically different at $p < 0.05$ by Tukey's test.

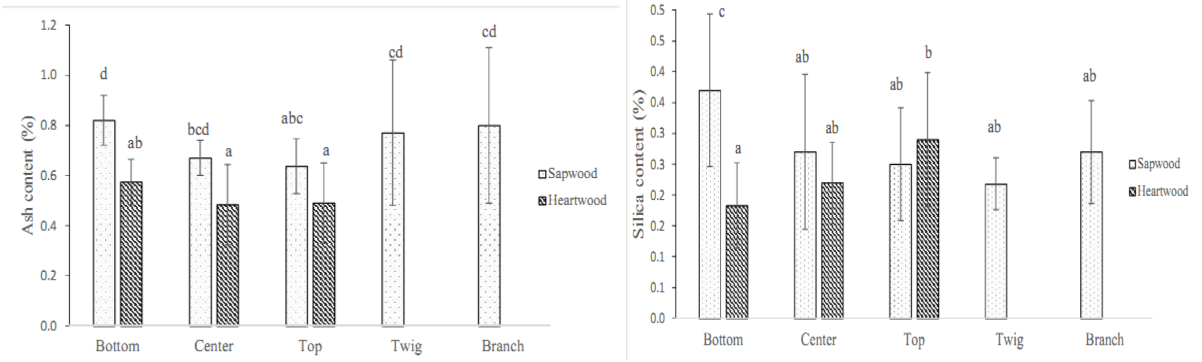


Figure 4. Ash and silica contents (%, extractive-free wood) of Jati Unggul Nusantara (Superior teak) wood by tree part. Average of 3 trees, with the standard deviation error bar. The same letters are not statistically different at $p < 0.05$ by Tukey's test.

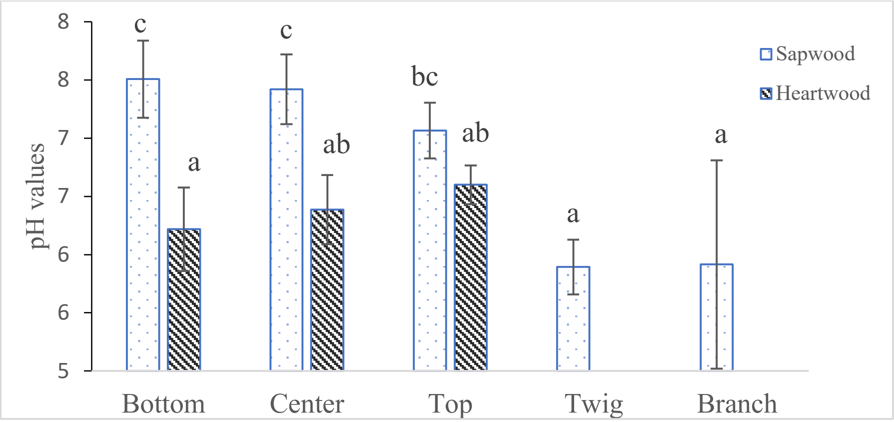


Figure 5. pH values of Jati Unggul Nusantara (Superior teak) wood by tree part. Average of 3 trees, with the standard deviation error bar. The same letters are not statistically different at $p < 0.05$ by Tukey's test.

Between the main stem and branch parts or the main stem and twig parts, there were significant differences in varied positions for all properties. The values of ethanol-toluene extractive, lignin, holocellulose, cellulose, hemicellulose, silica, and pH in the branches or twigs were generally lower than those in the main stem. The high ash content at the branch and twig parts is caused by the vicinity to the canopy as a place of photosynthesis where metals are necessary during the process. Between the twig and branch parts, statistical differences were observed in ethanol-toluene extractive, holocellulose, and hemicellulose contents, where the twig parts had higher levels in all the aforementioned contents.

Twigs and branches have been utilized for furniture making in the past few years. The different chemical properties between the twig and branch parts and the main stem should be noticed. However, information on twig chemistry is still limited. With regard to main stem and branch parts, these findings were different compared to earlier studies. Hassan *et al.* (2020) found that, in *Pinus halepensis* and *Eucalyptus camaldulensis*, compared to the stem wood, the contents of lignin, extractives, and ash of the branch wood were higher, but the contents of cellulose and hemicellulose were lower. In *Trema orientalis*, the branch samples consistently had lower cellulose and higher lignin and extractive contents than the stem samples (Jahan *et al.* 2010). A different pattern was observed in plum (*Prunus domestica*), where no significant differences between stem and branch samples in cellulose, lignin, and extractive contents were observed (Kiaei *et al.* 2014). The differences in species, tree age, and site were probably the reasons for the inconsistent patterns.

Comparison between Sapwood and Heartwood

By Tukey tests (Fig. 3~5), significant differences between the sapwood and heartwood in the main stem were observed in the contents of ethanol-toluene extractives (bottom and center parts), holocellulose (bottom part), cellulose (top part), lignin (center part), ash (bottom and center parts), and silica (bottom part), as well as in pH values (bottom and center parts). The heartwood part had higher values in ethanol-toluene extractive, holocellulose, and cellulose contents but lower in lignin, ash, silica, and pH values. Previous research on teak wood showed that there were no significant differences between sapwood and heartwood in holocellulose and α-cellulose contents (Miranda *et al.* 2011; Lukmandaru *et al*. 2016). Using 50~70-year-old teak from forests in East Timor, Miranda *et al.* (2011) observed no significant difference in lignin content between sapwood and heartwood (32.4% and 32.2%, respectively). It indicates that, in the case of juvenile wood, such as the JUN wood in this study, significant differences exist, but in mature wood, these differences are no longer visible.

The ethanol-toluene extractive content (Fig. 2) showed significant differences between sapwood and heartwood except at the bottom and center parts. A previous work observed that superior teak wood (11 years, JPP) showed significant differences in ethanol soluble extractives in the sapwood and heartwood in all vertical parts of the stem (Zulkahfi *et al.* 2020). The average value of ethanol-toluene extractive content was greater in the heartwood than in the sapwood in Gunungkidul community forest teak (Lukmandaru *et al.* 2016), Indonesian plus teak (Rudman *et al.* 1966), and Panama teak (Windeisen *et al*. 2003). Research related to hot-water solubility found no significant difference between the sapwood and the heartwood. This result is different from the earlier study by Lukmandaru *et al.* (2016), which showed significant differences between the sapwood and the inner and outer heartwood. It is assumed that these differences in value were influenced by the environmental conditions of the stand under observation, the age of the JUN wood, and the extraction method used.

The ash and pH values (Fig. 4 and 5) showed significant differences between the sapwood and the heartwood at the bottom and center parts. These differences might be caused by the age and condition of the stand. Furthermore, there were both similarities and differences between the current patterns and the patterns of plantation teak in ash, silica, and pH values (Lukmandaru and Hidayah 2016; 2018).

Conclusions

The wood properties of JUN were investigated for the tree parts. Among the main stem parts, significant differences were found in all properties except for hot-water solubility. The values in the branches or twigs were generally lower than those of main stem for ethanol-toluene extractive, lignin, holocellulose, cellulose, hemicellulose, silica, and pH values. Compared to the branch, twig parts had higher levels in ethanol-toluene extractive, holocellulose, and hemicellulose. Compared the sapwood part, the heartwood part had higher values in ethanol-toluene extractive, holocellulose, and cellulose contents but lower in lignin, ash, silica, and pH values. To establish the differences among the tree parts, investigations with more individual samples as well as young teak samples from various locations will be necessary in the next work due to high standard deviation in most parameters in this experiment.

Acknowledgement

This study was financed by DPP Grant 2020 - Faculty of Forestry Universitas Gadjah Mada.

References

Amin, Y.; S.A., Danang, Wahyuni, I., Sukma, S.K & Damayanti, R. (2013). Anatomical characteristics and chemical properties of the branch-wood of *Schizolobium amazonicum* Ducke species and its potential uses. *Indonesian* Journal of Forestry Research 10(2): 119–125.

Anonymous. (2011). Jati Unggul Nusantara (JUN). http://www.jatijun.com.

ASTM. 2002. Annual Book of ASTM Standards. Section Four Construction Volume 04.10 Wood. West Conshohocken, PA.

Bhat, K.M.; P.B. Priya; P Rugmini. 2001. Characterisation of juvenile wood in teak. Wood Science and Technology 34: 517–532.

Browning, B.L. 1967. Methods of Wood Chemistry Vol. I. Interscience Publishers, A Division of John Wiley and Sons, Inc. New York.

Dewan Standarisasi Nasional. 1989. SNI 14-1031-1989. Cara uji kadar abu, silika dan silikat dalam kayu dan pulp kayu. Dewan Standarisasi Nasional. Jakarta

Efansyah, M.N.; M.H. Bintoro; W.H. Limbong, 2012. Prospek usaha bagi hasil penanaman Jati Unggul Nusantara (Studi kasus pada Koperasi Perumahan Wanabhakti Nusantara di Kabupaten Bogor). Manajemen IKM 7(1): 64–73.

Hachmi, A.; A.A. Moslemi1990. Effect of wood pH and buffering capacity on wood-cement compatibility. Holzforschung 44:425-430.

Hassan, K.T.; E.A.E. Kandeel; I.E.A. Kherallah; H.A. Abou-Gazia; F.M.M. Hassan. 2020. *Pinus halepensis* and *Eucalyptus camaldulensis* grown in Egypt - A comparison between stem and branch properties for pulp and paper making BioResources 15(4):7598-7614

Hse, C.; M. Kuo. 1988. Influence of extractives on wood gluing and finishing - a review, Forest Products Journal 38(1): 52-56.

Jahan; M.S.; N. Chowdhury; Y.Ni. 2010. Effect of different locations on the morphological, chemical, pulping and papermaking properties of *Trema orientalis* (Nalita). Bioresource Technology 10: 1892-1898.

Iswanto, A.H.; F. Tarigan; A. Susilowati; A. Darwis; W. Fatriasari. 2021. Wood chemical compositions of raru species originating from Central Tapanuli, North Sumatra, Indonesia: Effect of differences in wood species and log positions. Journal Korean Wood and Science Technology 49(5):416-429.

Kanazawa, H.; T. Nakagami; K. Nobashi; T. Yokota. 1978. Studies on the gluing of the wood Articles. XI. The effects of teak wood extractives on the curing reaction and the hydrolysis rate of the urea resin. Mokuzai gakkaishi 24: 55-59.

Kiaei M.; M. Tajik; R. Vaysi. 2014. Chemical and biometrical properties of plum wood and its application in pulp and paper production. Maderas Ciencia y tecnología 16(3): 313-322.

Lukmandaru, G.; A.R. Mohammad; P. Wargono; V.E. Prasetyo. 2016. Studi mutu kayu jati di hutan rakyat Gunungkidul. V. Sifat kimia kayu. Jurnal Ilmu Kehutanan 10(2):108-118.

Lukmandaru, G.; R.N. Hidayah. 2017. Studi mutu kayu jati di hutan rakyat Gunungkidul. VI. Kadar zat anorganik dan keasaman. Jurnal Ilmu Kehutanan 11(1): 63-75.

Lukmandaru, G.; R.N. Hidayah. 2018. Measurements of inorganic materials and acidity in plantation teakwood. Wood Research Journal 9(2):35-44

Maloney, T.M. 1993. Modern particleboard and dry-process fiberboard manufacturing (updated edition). Miller Freeman, San Fransisco.

Maulida, F.; K.B. Meiganati; M. Maslahat. 2020. Komponen kimia kayu trubusan Jati Unggul Nusantara (*Tectona grandis* Linn.f.) pada bagian pangkal, tengah dan ujung. Jurnal Sains Natural 10(2): 55-60.

Miranda, I.; V. Sousa; H.Pereira. 2011. Wood properties of teak (*Tectona grandis*) from mature unmanaged stand in East Timor. Journal of Wood Science 57(3): 171–78.

Ona, T.; T. Sonoda, K. Ito, M. Shibata. 1998. Relations between various extracted basic densities and wood chemical components in *Eucalyptus globulus*. Journal of Wood Science 44: 165-168.

Rahman, F.; G. Lukmandaru. 2022. Extractive contents of the juvenile stemwood and bark of of teak. Wood Research 67(1):96-108.

Rahman, W.M.N.W.A.; N.Y.M. Yunus; J. Kasim; N.S.M. Tamat. 2018. Effects of tree portion and radial position on physical and chemical properties of kelampayan (*Neolamarckia cadamba*) wood. BioResources 13(2): 4536-4549.

Rudman, P.; H. J. Gay; E.W.B. Da Costa. 1967. Wood quality in plus trees of teak (*Tectona grandis* L.f.): An assessment of decay and termite resistance. Sylvae Genetica 16: 102–5.

Technical Association for the Pulp and Paper Industries. 1992. TAPPI Test Method T 222 os-74. TAPPI Press. Atlanta.

Rowell, R.; R. Pettersen; J. S., Han, Rowell, J.S., & Tshabala, M.S. (2005). Cell wall chemistry. In: *Handbook of wood chemistry and wood composites*. Rowell, R. (Ed). CRC Press. Boca Raton London New York Washington, D.C.

Rizanti, D.E., Wayan, D., Beatrice, G., Andre, M., Stephane, D., Hubert, C., Christiane, G., Eric, G., Phila, R., Sari, R.K., Syafii, W., Rozi, M., & Philippe, G. (2018). Comparison of teak wood properties according to forest management: Short versus long rotation. *Annals of Forest Science* 75: 39

Samariha, A.; M. Kiaei. 2011. Chemical composition properties of stem and branch in *Alianthus altissima* wood. Middle-East Journal of Scientific Research 8(5): 967–70.

Shmulsky, R.; P. D. Jones. 2011. *Forest Products and Wood Science: An introduction*. Sixth edition.

Syahidah, H.; A.D. Yunianti. 2007. Kandungan kimia dan dimensi serat akar, cabang dan batang bagian atas kayu gmelina dan kayu jati di hutan rakyat Sulawesi Selatan. Jurnal Perennial 3(1):11-14.

Thulasidas, P.; H. Bailleres. 2017. Wood quality for advanced uses of teak from natural and planted forests. IUFRO World Series 36:73-81. Windeisen, E.; A. Klassen, G. Wegener 2003. On the chemical characterization of plantation teakwood (Tectona grandis L.) from Panama. Holz als Roh-und Werkstoff 61: 416–418.

Zulkahfi; D., Irawati; T. Listyanto; D. Rodiana; G. Lukmandaru. 2020. Kadar ekstraktif dan sifat warna kayu Jati Plus Perhutani umur 11 tahun dari KPH Ngawi. Jurnal Ilmu Kehutanan 14: 213-227.

Ganis Lukmandaru, Ardhan Wira Pratama, JP Gentur Sutapa, Sigit Sunarta, Denny Irawati, Rini Pujiarti, Rizki Arisandi
Department of Forest Products Technology, Faculty of Forestry, Gadjah Mada University
Jl. Agro, Bulaksumur, Sleman
Tel : 0274-901420 ;
Fax : 0274-550541
Email : glukmandaru@ugm.ac.id

A Review: The Soluble Sugars Involved in The Process of Heartwood Formation

Rizki Arisandi

Abstract

This study aims to examine the soluble sugars involved in the process of heartwood formation using 87 journal articles and non-articles. The data used was collected through an online search with 5 keywords, namely “heartwood formation”, “reserve material of heartwood”, “non-structural carbohydrate of heartwood”, “starch of heartwood” and “sucrose of heartwood”. By removing irrelevant papers, 44 suitable journal articles and non-articles were found. Studies showed that monosaccharide fraction was the largest group compared to the other fractions as well as dominated by the hexose group. In addition, several species such as *Swietenia mahagoni, Pinus sylvestris, Betula pendula,* and *Fagus sylvatica* were detected more complete than other species in the fraction of soluble sugars (monosaccharides, disaccharides, trisaccharides, tetrasaccharides, alditol, and cyclitol). Based on previous studies, the contents of glucose and sucrose compounds were the major compound and plays an important role in storing energy reserves and the process of heartwood formation. Meanwhile, the levels of arabinose and galactose were the minor components in several species. Within tree variation, soluble sugars drop from the sapwood to the heartwood and from the top to the bottom of the tree. Subsequently, the alditols and cyclitols fractions need special issues, especially for tropical species related to their role.

Keyword: Heartwood formation, reserve material, non-structural carbohydrate, starch, sucrose

Introduction

In most tree species, the xylem consists of two histologically similar but physiologically different wood zones (sapwood and heartwood). Sapwood (the outer wood zone) is composed of physiologically active, live cells and reserve substances, and the external rings transport water with minerals from roots to the cambium and assimilatory organs. Meanwhile, heartwood (the inner wood zone) is physiologically inactive and does not participate in the conduction of water (Nawrot *et al.* 2008).

Heartwood formation is an important process in perennial plants as trees. It is the ultimate process leading to the death of living sapwood tissues due to internal phenomenon depending on the cycle of tree life involving both water and mechanical gradients (Berthier *et al.* 2001). Physiological processes such as xylem dehydration (Kuroda *et al.* 2009), depletion of storage compounds (Magel *et al.* 1994; Piispanen and Saranpaa 2001), accumulation of heartwood substances (Magel *et al.* 1991; Nakada and Fukatsu 2012), and programmed cell death (Nakaba *et al.* 2012), often accompanies the process of the heartwood formation. In addition, there is also changes in the structure of the cell wall (Nakada and Fukatsu 2012; Song *et al.* 2014). The substance deposited in the heartwood is the most important investment because of its effect on the color value (aesthetics) and natural durability. Secondary metabolism in trees produces heartwood substances whose substrates come from nonstructural carbohydrate metabolism (NSC) (Cui *et al.* 2020).

Reserve materials are present in the wood as lipids and NSC. Storage of NSC is very important for woody species. These reserves were used in long-lived organisms for their perennity by fighting against biotic and abiotic stress, including drought, disturbance, and pests (Dietze *et al.* 2014). NSC play an important role such as carbon providing for energy metabolism and osmoregulation, nutrient transport, and the biosynthesis of toxic extractives for defense mechanisms (Kampe and Magel 2013).

During the formation of the heartwood secondary components, NSC is the main photosynthetic storage compound which is transported inwards through the ray parenchyma cells (Hillis 1987). The appearance of cell death in the transition zone (sapwood-heartwood) begins with depletion of storage substances based on increased endoamylolytic activity. In all sapwood part of the trees shows a more or less even distribution of all NSC within the sapwood. However, the concentrations of starch and soluble sugars decreased steadily with increasing age of the sapwood to the heartwood boundary. In most species, the NSC is almost absent in the heartwood. This is found for both softwood and hardwood (Magel and Holl 1993; De Jardin *et al.* 1997). Hillis (1987) stated that the composition and distribution of NSC depend on the growing location, wood species, within tree variation, and genetic factors. Furthermore, NSC consists of starch and soluble sugars, such as sucrose, fructose, glucose, arabinose, galactose, and stachyose, etc., (Cui *et al.* 2020). Soluble sugars that are usually involved in the heartwood formation process are monosaccharides (e.g. glucose, fructose, mannose, xylose and galactose), disaccharides (sucrose), and a small number of trisaccharides (e.g. raffinose) and tetrasaccharides (e.g. stachyose). This review will be only focus on the soluble sugars involved in the process of heartwood formation.

This study aims to carry out a literature review of the soluble sugars involves in the heartwood formation. In this study, articles and non-articles were investigated through online searches. The keywords used to search for journals and non-journal articles including “heartwood formation”, “reserve material of heartwood”, “non-structural carbohydrate of heartwood”, “starch of heartwood” and “sucrose of heartwood”. Furthermore, a total of 87 journal articles and non-articles were found. However, only 44 suitable papers were used for review (Figure 1).

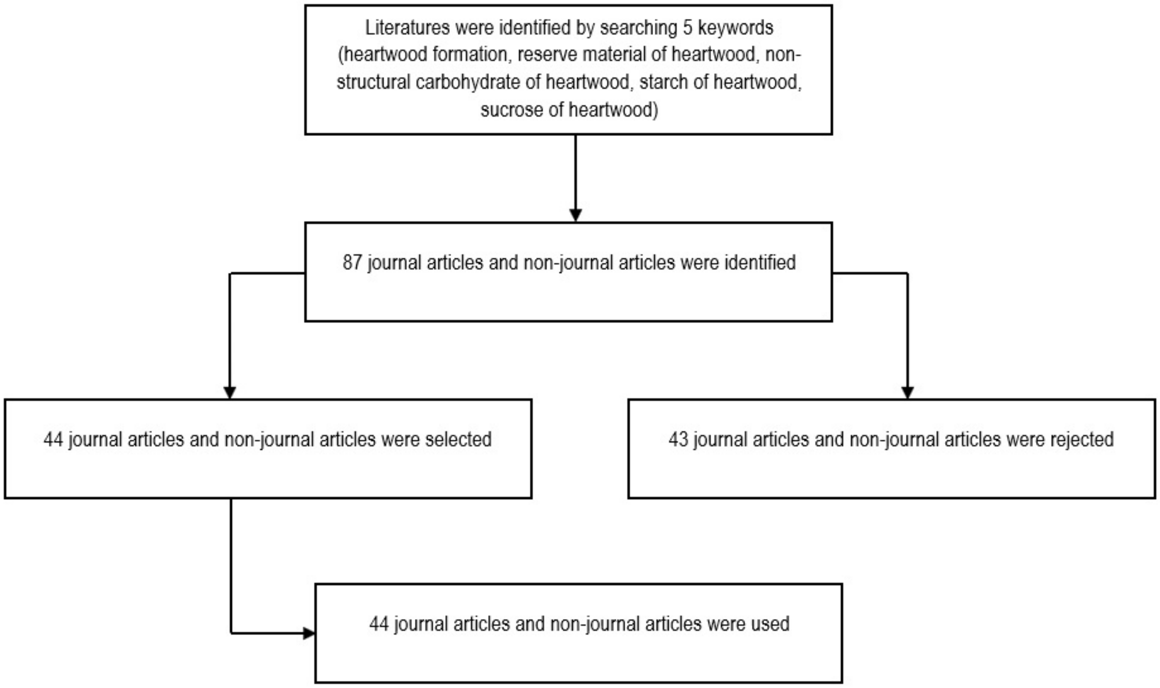


Figure 1. Review processes for this study

Soluble Sugars in Some Species

In mahogany species such as *Swietenia mahagoni*, fractions of monosaccharides, cyclitols, alditols and disaccharides were found, while in *Swietenia macrophylla* only monosaccharide was detected. The similar pattern was also found in same families (*Meliaceae*), such as in seedling of *Cedrela fissilis* (Aragao *et al.* 2015), stem bark of *Azadirachta indica* (Chitra *et al.* 2017) and in wood of *Acacia melanoxylon* (Lourenco *et al.* 2008). Furthermore, in other species like *Quercus faginea* and *Trema orientalis*, mostly monosaccharide groups were also found (Jahan and Mun, 2003; Miranda *et al.* 2017). On the other hand, monosaccharide and disaccharide groups were obtained in,, for instance, *Tectona grandis, Robinia pseudoacacia,* and *Schinopsis balansae* (Magel *et al.* 1994; Streit and Fengel 1994; Niamke *et al.* 2010, 2011). Furthermore, in the *Pinus species*, there were several slightly different patterns. Saranpaa and Holl (1989) and Funda *et al.* (2020) reported that monosaccharides, disaccharides, trisaccharides, and tetrasaccharides fractions were detected in *Pinus sylvestris*. Meanwhile, Turfan *et al.* (2018) stated that monosaccharides (glucose) and disaccharides (sucrose) groups were detected in *Pinus nigra.* On the other hand, it was just monosaccharides observed in *Pinus radiata* (Uprichard and Lloyd 1980; Berrocal *et al.* 2004). Subsequently, in *Delbergia odorifera* species, monosaccharides, disaccharides (sucrose) and tetrasaccharides (stachyose) were detected. The similar trend was also shown in *Betula pendula* detecting monosaccharides, trisaccharides (raffinose) disaccharides (sucrose), cyclitols (myo-inositol) and in *Fagus sylvatica* namely, monosaccharides, disaccharides, trisaccharides (raffinose), tetrasaccharides (stachyose), alditols, and cyclitols (inositol). The complete soluble sugar groups and compounds in the several species mentioned previously are presented in Table 1.

Table 1. Soluble sugars in some species both in hardwood and softwood (stem wood part)

Wood categories	Species	Soluble sugars	References
Hardwoods	*Swietenia mahagoni*	monosaccharides (glucose, fructose, mannose, arabinose, galactose, xylose), cyclitols (myo-inositol and neo-inositol), alditols (meso-erytrithol, arabitol, mannitol), disaccharides (sucrose)	Arisandi *et al.* 2022
	Swietenia macrophylla	monosaccharides (glucose, mannose, arabinose, galactose, xylose, rhamnose)	Rutiaga-Quiñones *et al.* 1998
	Acacia melanoxylon	monosaccharides (glucose, mannose, arabinose, galactose, xylose)	Lourenco *et al.* 2008
	Dalbergia odorifera	monosaccharides (glucose, fructose, arabinose, galactose) disaccharides (sucrose), tetrasaccharides (stachyose)	Ciu *et al.* 2020
	Tectona grandis	monosaccharides (glucose, fructose), disaccharides (sucrose)	Niamke *et al.* 2010, 2011
	Robinia pseudoacacia	monosaccharides (glucose, fructose), disaccharides (sucrose)	Magel *et al.* 1994
	Schinopsis balansae	monosaccharides (glucose, fructose, mannose, galactose, xylose, rhamnose), disaccharides (sucrose)	Streit and Fengel 1994
	Betula pendula	monosaccharides (glucose, fructose, raffinose, disaccharides (sucrose), cyclitols (myo-inositol)	Piispanen and Saranpaa 2001
	Quercus faginea	monosaccharides (glucose, arabinose, mannose, galactose, xylose, rhamnose)	Miranda *et al.* 2017
	Fagus sylvatica	monosaccharides (glucose, fructose, arabinose, mannose, galactose, xylose, rhamnose), disaccharides (trehalose, saccharose), trisaccharides (raffinose), tetrasaccharides (stachyose), alditols (erythritol, glycerol, arabitol and mannitol, sorbitol, xylitol), cyclitols (inositol)	Dietrichs 1964; Kubel *et al.* 1988; Irmouli *et al.* 2002; Zule and Moze 2003; Vek *et al.* 2014, 2016
	Trema orientalis	glucose, arabinose, mannose, galactose, xylose	Jahan and Mun 2003
Softwood	*Pinus radiata*	monosaccharides (glucose, mannose, arabinose, galactose, xylose)	Uprichard and Lloyd 1980; Berrocal *et al.* 2004
	Pinus sylvestris	monosaccharides (glucose, fructose, mannose, arabinose, galactose, xylose), disaccharides (sucrose), trisaccharides (raffinose), tetrasaccharides (stachyose)	Saranpaa and Holl 1989; Funda *et al.* 2020
	Pinus nigra	monosaccharides (glucose), disaccharides (sucrose)	Turfan *et al.* 2018

In the compound levels, glucose is the largest component in *Swietenia mahagoni, Swietenia macrophylla, Acacia melanoxylon, Pinus radiata, Pinus sylvestris, Dalbergia odorifera, Tectona grandis, Schinopsis balansae, Quercus faginea, Fagus sylvatica,* and *Trema orientalis.* However, the opposite pattern was found in sucrose levels found in *Robinia pseudoacacia, Pinus sylvestris* or *Fagus sylvatica.* The low sucrose content occurs due to reduced transport in the phloem (Niamke *et al.* 2010). The relatively low phloem activity can be associated to the harvest season (Nobuchi *et al.* 2005) which corresponds to the begin of the dry season and shows high enzymatic hydrolysis of sucrose to fructose and glucose (Magel *et al.* 1994). Meanwhile, species such as *Pinus nigra* (Turfan *et al.* 2018) and *Robinia pseudoacacia* (Magel *et al.* 1994) are detected the highest sucrose compared to glucose. The low glucose and high sucrose content indicate that glucose are metabolized to sucrose and used to increase osmotic potential (Turfan *et al.* 2018). In addition, levels of arabinose and galactose are generally low in several species, especially in the species *Dalbergia odorifera, Swietenia mahagoni, Acacia melanoxylon, Pinus radiata* and *Pinus sylvestris* (Uprichard and Lloyd 1980; Saranpaa and Holl 1989; Berrocal *et al.* 2004; Lourenco *et al.* 2008; Funda *et al.* 2020; Ciu *et al.* 2020; Arisandi *et al.* 2022). This might be related to their function and characteristics (Hansen *et al.* 1997). Saranpaa and Holl (1989) reported that these sugar compounds were the smallest storage materials. Therefore, it was the most easily utilized carbohydrates in trees. With regard to hexose and pentose compositions, hexoses were greater than pentoses. Important metabolic intermediates i.e., hexoses (particularly glucose and fructose) can be used to form storage groups of carbohydrates (particularly starch, which is a glucose polymer), and other disaccharide compounds for transport throughout the body from organisms (Sage 2008).

At some species were also detected not only monosaccharide and disaccharide fractions, but also sugar alcohol group including alditols and cyclitols such as in *Swietenia mahagoni, Betula pendula*, and *Fagus sylvatica.* In mahogany, alditols and cyclitols have not known regarding their role in the heartwood, because they usually play a specific role in temperate species. For example, in *Betula* *platyphylla var. japonika* (Kasuga *et al.* 2007), where alditols and cyclitols play an important role for abiotic stress tolerance. They are primarily involved in tolerance to drought, salt stress, and low temperatures. Cyclitols and alditols in mahogany are involved in certain activities and functions which are tropical species. Magel *et al.* (2000) stated that alditol is involved in cell elongation, carbon transport and cryoprotection and is synthesized in appreciable quantities under specific metabolic conditions. Additionally, the biological functions of cyclotol differ from one another. Piispanen and Saranpaa (2001) mentioned that myo-inositol in silver birch serves as a reserve for oligosaccharide metabolism intermediates of the raffinose family, which is required during cold acclimatization in autumn. In mahogany, cyclitol can function as a metabolic reserve pool for the more active parts of the stem (wood cells near the cambium) (Piispanen and Saranpaa 2001). Several other species such as the heartwood of *Planchonella vitiensis* (Cambie *et al.* 1997) and *Sequoia sempervirens* (Anderson *et al.* 1968) and also in the stems of *Betula pubescens* and *Betula pendula* stems (Linberg *et al.* 1958), myo-inositol was detected.

In general, hardwood contains more pentosans (C-5 sugars) such as xylose and arabinose, while softwood contains more hexosans (C-6 sugars) such as mannose, glucose and galactose. Lachowicz *et al.* (2019) stated that softwood hemicellulose consists of pentosans and hexosans, but hardwood hemicellulose consists mainly of pentosans. In addition, the main differences between tropical and temperate species that NSC, starch and soluble sugars in tropical species during the dry season are generally higher than in temperate species such as in the species *Acer saccharum* and *Betula alleghaniensis* in North America (Gaucher *et al.* 2005). However, in the rainy season, the NSC content is relatively the same between tropical and temperate species (Würth *et al.* 2005; Myers and Kitajima 2007), for example, in the Panama area and temperate in East China (Chen *et al.* 2012; Zhang *et al.* 2013).

Within and Among Tree Variation

Lachowicz *et al.* (2019) stated that the relationship between wood properties and tree age has long been a topic of interest for many researchers. Berrocal *et al.* (2004) reported that the glucose and mannose contents increased, while the xylose, galactose, and arabinose concentrations decreased with tree age from 1 to 15 years in *Pinus radiata.* Rencoret *et al.* (2011) stated the content of arabinan, galactan, and mannan decreased, while the amount of glucan and xylan increased, with increasing age of *Eucalyptus globulus* trees from 1 month to 9 years. Other studies have also reported the effect of tree age on sugar components in several species such as *Eucalyptus globulus* and *Pinus radiata* (Uprichard and Lloyd 1980; Miranda and Pereira 2022).

NSC and lipids as reserve materials are transported from sapwood through ray parenchyma cells to supply the formation of secondary compounds in heartwood. These compounds are usually stored in the part of sapwood (Magel *et al.* 1994). Some NSC such as glucose, fructose and sucrose were detected in sapwood and their amount decreased from cambium to heartwood (Dietrichs 1968). Meanwhile, other sugars, namely xylose and arabinose were detected in *Quercus sp* heartwood and mannose Norway spruce heartwood.

Arisandi *et al.* (2022) reported that the main soluble monosaccharide sugars (glucose, fructose, mannose, and xylose) in *Swietenia mahagoni* accumulated in the sapwood and decreased progressively from the sapwood to the heartwood. This is indicated that these compounds are metabolized at the heartwood-sapwood boundary (Niamke *et al.* 2011). The high contents of the major components in sapwood could be a high demand for NSC which were degraded for energy and the carbon skeleton needed for metabolic activities such as photosynthesis, the formation of secondary metabolites (phenolics), and cell respiration (Datta and Kumar 1987; Magel *et al.* 1994). In addition, the heartwood-sapwood boundary proved to be the site where the highest NSC catabolic activity occurred (Magel *et al.* 2001). Transition of metabolic activity from sapwood to extractive forms of heartwood (phenolic compounds) from reserves (NSC) has also been reported (Datta and Kumar, 1987; Nobuchi *et al.* 1996). Niamke *et al.* (2011) found that NSC (starch, glucose, fructose and sucrose) decreased drastically from sapwood to heartwood in teak wood.

In the transition zone, the amount of starch can be hydrolyzed into glucose through the hydrolysis of the amyloglycosidase enzyme (Magel *et al.* 1997; Magel *et al.* 2001). In other species such as teak, the key enzymes for NSC catabolism are succinate-dehydrogenase and glucose-6-phosphate involved in reducing NSC and accumulates in the inner sapwood and the transition zone (Datta and Kumar 1987). In addition, several minor monosaccharide components (arabinose and galactose) are absent in heartwood. Similar results were found in *Dalbergia odorifera,* where this may be related to its characteristics and function (Cui *et al.* 2020). Saranpaa and Holl (1989) reported that monosaccharides are small storage materials and are the most easily used carbohydrates in trees.

Sucrose is one of the major carbohydrate components, but this compound is only found in a small proportion of heartwood, or not detect (Streit and Fengel, 1994; Magel *et al.* 1994; Niamke *et al.* 2011). Arisandi *et al.* (2022) reported that sucrose is low detected in mahogany sapwood. However, based on the results of the principal component analysis (PCA), the sucrose component was always found during the formation of heartwood. Therefore, this shows that the presence of sucrose is very important in the process of heartwood formation. The content of sucrose decreased from heartwood to sapwood at 4 years, but it was not found at 5 years old in heartwood part (Arisandi *et al.* 2022). Furze *et al.* (2018) stated that in the ray parenchyma cell, NSC can be transported in two opposite directions. It means that the NSC transported inward can be used as a substrate for secondary metabolism in the xylem, including for the synthesis of heartwood substances. While NSC transported outside retain the phloem "Leakage-retrieval Mechanism" (De Schepper *et al.* 2013). Magel *et al.* (1994) mentioned that the nature and radial distribution of carbohydrate reserves in wood indicated that the youngest zone of wood (outer sapwood) contains high concentrations of soluble sugars (sucrose, glucose and fructose predominately) and starch, whereas the heartwood part is almost absent of storage material. IAWA (1964) reported that in living trees, the sapwood contains cells, reserve materials, and is rich in nutrients that are attractive to biotropic pathogens. Meanwhile, heartwood is rich in secondary metabolites (flavanols and other phenolic compounds) to protect tree from wood destroying microorganism.

Conclusions

Soluble sugar is one of the nonstructural carbohydrate metabolism that play an important role in the process of heartwood formation. They are metabolized in the transition zone to produce substance heartwood products (phenolic compounds) during the process of the formation of heartwood. Apart from monosaccharide and disaccharide groups, several fractions of trisaccharides, tetrasaccharides, alditols, and cyclitols were also detected in *Swietenia mahagoni, Pinus sylvestris, Betula pendula,* and *Fagus sylvatica.* Glucose and sucrose were the largest compounds in several species in this study. Therefore, their role is very important in the process of heartwood formation. On the other hand, hexose group in the monosaccharide fraction is the largest group. Subsequently, further studies need to be carried out to examine the role of alditol and cyclitol compounds found in tropical species.

References

Arisandi, R.; S.N. Marsoem; G. Lukmandaru; J.P.G. Sutapa. 2022. Analysis of sugar components related to heartwood formation in young *Swietenia mahagoni* (L.) Jacq Trees. Journal of Wood Chemistry and Technology 42(3): 137-148.

Aragao, V.P.M.; B.V. Navarro; L.Z. Passamani; A.F. Macedo; E.S. Floh; V. Silveira; C. Santa-Catarina. 2015. Free amino acids, polyamines, soluble sugars and proteins during seed germination and early seedling growth of *Cedrela Fissilis Vellozo* (Meliaceae), An endangered hardwood species from the Atlantic forest in Brazil. Theoretical and Experimental Plant Physiology 27: 157–169.

Berrocal, A.; J. Baeza; J. Rodriguez; M. Espinosa; J. Freer. 2004. Effect of tree age on variation of *Pinus radiata* D. don chemical composition. Journal of the Chilean Chemical Society 49: 251–256.

Berthier, S.; A.D. Kokutse; A. Stokes; T. Fourcaud. 2001. Irregular heartwood formation in Maritime Pine (*Pinus pinaster* Ait.): Consequences for biomechanical and hydraulic tree functioning. Annals of Botany 87: 19–25.

Cambie, R.C.; N.G.A. Ser; T. Kokubun. 1997. Heartwood Constituents of *Planchonella vitiensis*. Biochemical Systematics and Ecology 25: 677–678.

Chen, T.; H. Pei; Y. Zhang; Q. Qian. 2012. Seasonal changes in non-structural carbohydrates and sucrose metabolism enzymes in two Sabina species. Acta Physiologiae Plantarum 34: 173–180.

Chitra, V.; V.K.N. Senthil; V.A. Rahul; S. Usha; G.R.A. Jeeva. 2017. Review on *Veppampattai chooranam* – A siddha single herbal formulation. World Journal of Pharmaceutical Research 6: 366–372.

Cui, Z.; X. Li; D. Xu; Z. Yang. 2020. Changes in non-structural carbohydrates, wood properties and essential oil during chemically-induced heartwood formation in *Dalbergia Odorifera*. Frontiers in Plant Science. https://doi.org/10.3389/fpls.2020.01161

Datta, S.K.; A. Kumar. 1987. Histochemical studies of the transition from sapwood to heartwood in *Tectona Grandis.* IAWA Journal 8: 363–368.

Dietrichs, H.H. 1964. Das verhalten von kohlenhydraten bei der holzverkernung. Holzforschung 18: 14–24.

Dietze, M.C.; A. Sala; M.S. Carbone; C.I. Czimczik; J.A. Mantooth; A.D. Richardson; R. Vargas. 2014. Non-structural carbon in woody plants. Annu. Rev. Plant Biol. 65: 667–687.

Funda, T.; I. Fundova; A. Gorzsás; A. Fries; H.X. Wu. 2020. Predicting the chemical composition of juvenile and mature woods in Scots pine (*Pinus sylvestris* L.) using FTIR spectroscopy. Wood Science and Technology 54: 289–311.

Furze, M.E.; S. Trumbore; H. Hartmann. 2018. Detours on the phloem sugar highway: stem carbon storage and remobilization. Curr. Opin Plant Biol. 43: 89–95.

Gaucher, C; S. Gougeon; Y. Maufette; C. Messier. 2005. Seasonal variation in biomass and carbohydrate partitioning of understory Sugar Aple (*Acer saccharum*) and Yellow Birch (*Betula alleghaniensis*) seedlings. Tree Physiology 25: 93–100.

Hillis, W.E. 1987. Heartwood and Tree Exudates. Springer-Verlag: Berlin.

Jahan, S.; S.P. Mun. 2003. Characterization of Nalita Wood (Trema orientalis) as a source of fiber for papermaking (Part I): Anatomical, morphological and chemical properties. Journal of Korea TAPPI 35(5): 72-79.

Kampe, A.; E. Magel. 2013. New insights into heartwood and heartwood formation. in cellular aspects of wood formation. Berlin, Heidelberg: Springer, pp. 71-95.

Kasuga, J.; K. Arakawa; S. Fujikawa. 2007. High accumulation of soluble sugars in deep supercooling Japanese White Birch xylem parenchyma cells. New Phytol. 174: 569–579.

Kubel, H.; G. Weissmann; W. Lange. 1988. Investigations on the cancerogenicity of wood dust-the extractives of Beech and Spruce. Holz Roh- Werkst. 46: 215–220.

Kuroda, K.; K. Yamashita; T. Fujiwara. 2009. Cellular level observation of water loss and the refilling of tracheids in the xylem of *Cryptomeria Japonica* during heartwood formation. Trees 23: 1163–1172.

Lachowicz, H.; H. Wroblewska; R. Wojtan; M. Sajdak. 2019. The Effect of tree age on the chemical composition of the wood of Silver Birch (*Betula pendula* Roth.) in Poland. Wood Science and Technology 53: 1135–1155.

Lindberg, B.; L. Selleby; P. Svendsås; K. Hartiala; S. Veige; E. Diczfalusy. 1958. Birch Wood Constituents I. Carbohydrates of Low Molecular Weight. Acta Chem. Scand.12: 1512–1515.

Lourenco, A.; I. Baptista; J. Gominho; H. Pereira. 2008. The Influence of heartwood on the pulping properties of *Acacia melanoxylon* wood. Journal of Wood Science 54: 464–469.

Magel, E.A.; A. Abdel-Latif; R. Hampp. 2001. Non-structural carbohydrates and catalytic activities of sucrose metabolizing enzymes in trunks of two *Juglans* species and their role in heartwood formation. Holzforschung. 55: 135–145.

Magel, E.A.; A. Drouet; A.C. Claudot; H. Ziegler. 1991. Formation of heartwood substances in the stem of *Robinia pseudoacacia* L. Trees 5: 203–207.

Magel, E.A.; W. Holl. 1993. Storage carbohydrates and adenine nucleotides in trunks of *Fagus sylvatica* L. in relation to discolored wood. Holzforschung 47: 19–24.

Magel, E.; C. Jay-Allemand; H. Ziegler. 1994. Formation of heartwood substances in the stemwood of *Robinia pseudoacacia* L. II. Distribution of nonstructural carbohydrates and wood extractives across the trunk. Trees 8: 165–171.

Magel, E.; W. Einig; R. Hampp. 2000. Carbohydrates in trees. Dev. Crop. Science 26: 317–336.

Myers, J.A; K. Kitajima. 2007. Carbohydrate storage enhances seedling shade and stress tolerance in a neotropical forest. Journal of Ecology 95: 383–395.

Miranda, I.; V. Sousa; J. Ferreira; H. Pereira. 2017. Chemical characterization and extractives composition of heartwood and sapwood from *Quercus faginea.* PLose One 12(6): 1-14.

Nakada, R.; E. Fukatsu. 2012. Seasonal variation of heartwood formation in *Larix kaempferi.* Tree Physiology 32: 1497–1508.

Nawrot, M.; W. Pazdrowski; M. Szymański. 2008. Dynamics of heartwood formation and axial and radial distribution of sapwood and heartwood in stems of European Larch (*Larix decidua* Mill.). Journal of Forest Science 54(9): 409–417.

Niamke, F.B.; N. Amusant; A.D. Kokutse; G. Chaix; J.P. Charpentier; A.A. Adima; S.C. Kati-Koulibaly; C. Jay-Allemand. 2010. Radial distribution of non-structural carbohydrates in Malaysian teak. International Journal of Biological and Chemical Sciences 4: 710–720.

Niamke, F.B.; N. Amusant; J.P. Charpentier; G. Chaix; Y. Baissac; N. Boutahar; A.A. Adima; S. Kati-Coulibaly; C. Jay-Allemand. 2011. Relationships between biochemical attributes (non-structural carbohydrates snd phenolics) and natural durability against fungi in dry Teak wood (*Tectona Grandis* L. F.). Annals of Forest Science 68: 201–211.

Nobuchi, T.; N. Okada; M. Nishida; S. Siripatanadilok; T. Veenin; T.L. Tobing; M.H. Sahri. 2005. Some characteristics of wood formation in Teak (*Tectona Grandis*) with special reference to water conditions. in quality timber products of Teak from sustainable forest management, Bhat, K. M., Nair, K. K. N., Bhat, K. V., Muralidharan, E. M., Sharma, J. K., Eds; Proceedings of International Conference on Quality Timber Products of Teak from Sustainable Forest Management. India, pp. 495–499.

Piispanen, R.; P. Saranpaa. 2001. Variation of non-structural carbohydrates in Silver Birch (*Betula Pendula* Roth) wood. Trees 15: 444–451.

Rencoret, J.; A. Gutierrez; L. Nieto; J. Jimenez-Barbero; C.B. Faulds; H. Kim; J. Ralph; A.T.; Martinez; J.C. del Rio. 2011. Lignin composition and structure in young versus adult *Eucalyptus globulus* plants. Plant Physiology 155: 667–682.

Rutiaga-Quinones, J.G.; E.W.C. Strobel. 1998. Polysaccharide von *Swietenia macrophylla* King. European Journal of Wood and Wood Product 56: 234.

Sage, R.F. 2008. Environmental effects on photosynthesis and primary productivity. Elsevier B.V. pp. 291-300.

Saranpaa, P.; W. Holl. 1989. Soluble carbohydrates of *Pinus sylvestris* L. sapwood and heartwood. Trees 3: 138–143.

Song, K.; Y. Yin; L. Salmén; F. Xiao; X. Jiang. 2014. Changes in the properties of wood cell walls during the transformation from sapwood to heartwood. Journal of Materials Science 49: 1734–1742.

Streit, W.; D. Fengel. 1994. On the changes of the extractive composition during heartwood formation in *Quebracho Colorado* (*Schinopsis balansae* Engl.). Holzforschung 48: 15–20.

Turfan, N.; M. Alay; T. Sariyildiz. 2018. Effect of tree age on chemical compounds of ancient anatolian Black Pine (*Pinus nigra* subsp. pallasiana) Needles in Northwest Turkey. iForest 11: 406-410.

Uprichard, J.M.; J.A. Lloyd. 1980. Influence of tree age on the chemical composition of Radiata Pine. New Zealand Journal of Forestry Science 10: 551–557.

Würth, M.K.R.; S. Peláez-Riedl; S.J. Wright; C. Körner. 2005. Non-structural Carbohydrate Pools in a Tropical Forest. Oecologia 143: 11–24.

Vek, V.; P. Oven; I. Poljanšek. 2016. Review on lipophilic and hydrophilic extractives in tissues of common Beech. Drvna Industrija 67(1): 85-96.

Vek, V.; P. Oven; T. Ters; I. Poljansek; B. Hinterstoisser. 2014. Extractives of mechanically wounded wood and knots in Beech. Holzforschung 68: 529–539.

Zhang, H.; C. Wang; X. Wang; F. Cheng. 2013. Spatial variation of non-structural carbohydrates in *Betula platyphylla* and *Tilia amurensis* stems. Chinese Journal of Applied Ecology 24: 3050–3056.

Zule, J.; A. Moze. 2003. GC Analysis of extractive compounds in Beech Wood. Journal of Separation Science 26: 1292–1294.

Rizki Arisandi
Department of Forest Products Technology, Faculty of Forestry, Universitas Gadjah Mada, Jl. Agro No.1, Bulaksumur, Yogyakarta 55281, Indonesia
E-mail: rizki.arisandi@mail.ugm.ac.id

Effect of Maleic Acid and Glycerol Concentrations on the Characteristics of Glycerol Ester of Maleic Rosin

Rafli Nur Ramadhani, Ganis Lukmandaru, Sigit Sunarta, Widiyatno

Abstract

Gum rosin, distilled from the resin of pine trees (*Pinus merkusii*), is highly susceptible to degradation and oxidation. To maintain its quality, modification by fortification among other methods should be made where a stable product named glycerol ester of maleic rosin (GEMR) is produced. In the experiment reported in this paper, rosin was prepared on the laboratory scale. Fortification was performed using maleic acid of varied concentrations (8%, 10%, and 12%, w/w), followed by mixing with glycerol through an esterification process (10%, 12%, and 14%, w/w). The average yield of GEMR produced was in the range from 67.11% to 79.30%. The average softening point, acid number, and ash content were in the ranges of 91.67–120.67°C, 5.84–9.82 mg KOH/g, and 5 x 10^{-3}–14.7 x 10^{-3}%, respectively. No significant effect of concentration was observed on yield and acid number. The GEMR product was completely soluble in toluene at all concentration levels. It was found that the increase of glycerol portion affected the properties of GEMR, i.e., lower ash content and acid number, while higher softening point. Based on the acid number, solubility in toluene, and softening point values, the GEMR obtained in this research fulfilled the requirements of Chinese GEMR standards.

Keywords: gum rosin, modified rosin, fortification, esterification, acid number

Introduction

Gum rosin is a solid material produced by distilling pine oleoresin, obtained by tapping the stem of pine trees. Indonesia is the third biggest gum rosin producer in the world after Brazil and China. In Indonesia, the total production of gum rosin reached ± 66 tons/year, ± 80% of which was exported to various countries (Perhutani 2018). In practice, before shipping, degradation of gum rosin (non-modified) usually occurs during the stockpiling stage. One of the weaknesses of non-modified gum rosin is that it is prone to oxidation in an open space. Such a condition causes rosin to be susceptible to crystallizing from a solid form and to easily react with heavy metal salts, especially in varnish products (Khadijah and Chumaidi 2022). Therefore, gum rosin modification is necessary to generate a stable product. One of the methods of gum rosin modification is to mix glycerol and maleic acid to produce glycerol ester of maleic rosin (GEMR).

Maleic rosin can be used as a mixture for the wood composite industry (Rongxian and Wen 2009). It is a derivate product produced by fortification using maleic acid. Meanwhile, glycerol ester is one of rosin derivatives produced by esterification (Prasetyo *et al.* 2012). Glycerol and maleic acid serve as agents in the modification process. In a previous work, the effect of maleic acid concentrations on maleic rosin was observed (Wiyono 2007). Esterification of maleic rosin was conducted to find out the optimum cooking conditions (Prakoso *et al.* 2021). In this study, rosin modification was carried out using an esterification process, which changed the reaction between carboxylic acid and alcohol or glycerol into GEMR, releasing water (H_2O) in the process.

Materials and Methods

Oleoresin Distillation

The starting gum rosin was obtained from *Pinus merkusii* oleoresin tapping (drilling method) in a forest stand at RPH Samudra, BKPH Lumbir, KPH West Banyumas. Hydro-distillation (100 g of oleoresin) was performed on the laboratory scale with a three-necked flask (300 mL) above the heating mantle. Heating was carried out with a resin-water ratio of 1:1.5 at a temperature of 150°C for approximately 2 hours. The obtained gum rosin (control) was then tested for its physicochemical properties.

Fortification Process

Rosin (30 g) was transferred to a distillation flask and heated at an initial temperature of 150°C. The temperature was then increased to 200°C until the rosin melted. Maleic acid (technical grade) was then mixed at different concentrations based on the rosin initial weight (8%, 10%, and 12%, w/w) to obtain a dark homogenous mixture. The temperature was maintained at 200°C for approximately 1 hour. After that, maleic rosin acid or maleic rosin mixture was obtained.

Esterification Process

An esterification process was carried out by further heating the obtained maleic rosin at 280°C to produce

GEMR. Glycerol (technical grade) was added at various concentrations based on the rosin initial weight (10%, 12%, and 14%, w/w). The temperature was maintained at 280°C for approximately 2 hours. The mixture was stirred to acquire a homogeneous material. The obtained mixture was then dissolved in toluene solution to remove the remaining maleic acid. Water was added, and the mixture was transferred to a separating funnel and shaken. After that, the layer of GEMR was poured to a container gradually. The yield (%) was calculated based on the initial gum rosin weight.

Physico-chemical Properties

The softening point (ring and ball apparatus), acid number, solubility in toluene, and ash content were measured according to the SNI 7636:2020 standard. These measurements were carried out with five replications.

Data Analysis

The variation of physico-chemical properties of rosin were analyzed by two-way analysis of variance (ANOVA) with general linear model procedure followed by Tukey's (HSD) post-hoc test (*p* = 0.05). All statistical calculations were conducted using SPSS-Win 18.0.

Results and Discussion

Rosin Characteristics

The rosin produced by hydro-distillation method was tested according to the Indonesian National Standard (SNI 7636:2020). The results of the measurement of rosin physico-chemical properties are presented in Table 1. Color measurement was carried out visually to the limitations of testing equipment in the laboratory and by comparison to commercial gum rosin obtained from PGT Cimanggu. Based on the color observed (Fig. 1), the rosin was designated to be of Water White (WW) quality.

The softening point was determined to measure the turpentine content remaining in the rosin. The obtained softening point value (75.07°C) did not fall in the standard range of WW quality, causing the rosin to be classified as N quality rosin. Technically, the higher the softening point, the smaller the turpentine content remaining in the rosin. Meanwhile, the acid number was 190.49 mg KOH/g, which fell in the standard range (160~200 mg KOH/g).

Ash content testing was carried out to indicate the level of impurities contained in the control gum rosin. The ash content (9×10^{-3}%) was within the standard range for WW class rosin. It is expected that lower ash content would produce esters of better quality in the gum rosin (Santosa 2010). This finding was supported by the fact that the rosin tested here was completely dissolved in toluene. Technically, solubility in toluene indicates the presence of foreign matters in rosin.

Table 1. Physico-chemical properties of control gum rosin

No	Assesments	Value
1	Yield (%)	63.66
2	Solubility in toluene (1:1)	dissolved
3	Acid number (mg KOH/g)	190.49
4	Softening point (°C)	75.07
5	Ash content (%)	9×10^{-3}

Figure 1. Control gum rosin

Characteristics of Glycerol Ester of Maleic Rosin

The physico-chemical properties of the GEMR obtained are presented in Table 2. The average yield value of the GEMR was in the range from 67.11% to 79.30%. The highest yield (79.30%) was found with maleic acid and glycerol, each at a 12% concentration. However, based on the results of the analysis of variance, there was no significant effect of concentrations on the yield. This indicates that the portion range used in this experiment did not affect the yield. Therefore, it becomes necessary to explore the optimum concentrations in the next trials.

The average softening point ranged from 91.67ºC to 120.67ºC. The results of the analysis of variance showed that the interaction between the amounts of glycerol and maleic acid had a significant effect (*p* = 0.008). It was observed that the addition in the portion of maleic acid from 8% to 12% increased the softening point. On the contrary, the increase in the portion of glycerol until 14% decreased the softening point. The highest softening point value (120.67ºC) was obtained from the interaction between 12% glycerol and 10% maleic acid. Based on the Tukey's test results, the combination of glycerol at the smallest percentage of 10% and maleic acid at 12% gave a value of 104.33ºC, which was not significantly different from the highest value. The lowest softening point value of GEMR was found by combining 8% maleic acid and 10% glycerol. The higher the contents of fat, wax, and oil in rosin, the lower the softening point (Coppen and Hone 1995). This indicates that the gum rosin derivates still contained traces of foreign matters during the washing process.

Table 2. Average values of physico-chemical properties and solubility in toluene of glycerol ester of maleic rosin

Concentration of Glycerol (%)	Concentration of Maleic Acid (%)	Yield (%)	Softening Point (^{o}C)	Acid Number (mgKOH/g)	Ash Content (×10^{-3} %)
10	8	67.11	91.67 ^{a}	8.42	8.7
	10	74.87	93 ^{ab}	7.71	11.3.
	12	78.27	104.33 ^{abc}	9.82	14.7
	Average	73.42 ± 5.72	96.33 ± 6.96	8.65 ± 1.07	11.6 ± 3
12	8	76.69	106.67 ^{abc}	7.95	7.7
	10	78.09	120.67 ^{c}	7.48	11
	12	79.3	105.33 ^{abc}	9.12	13.3
	Average	78.03 ± 1.31	110.89±8.49	8.18±0.84	10.7 ± 2.9
14	8	73.42	104.67 ^{abc}	7.71	5
	10	74.44	104 ^{abc}	5.84	9.5
	12	75.42	113.33 ^{bc}	8.88	14.3
	Average	74.43 ± 1	107.33 ± 5.21	7.48 ± 1.53	9.6 ± 4,7

Remarks: The same letters on the same column are not statistically different at $P < 0.05$ by Tukey's test.

The acid number indicates the amount of free fatty acids in rosin products (Wiyono 2007). Gum rosin contains 90% resin acids and 10% non-acids; carboxylic acids mostly take the form of abietic acids (Hiller *et al.* 2007; Fiebach 1993). Reducing the acid number would improve the quality of the GEMR product. Less unreacted carboxylic acids would remain, and more polyester would be formed. Therefore, the product would become more stable and not easily damaged when exposed to alkaline chemicals (Purnavita *et al.* 2017). The results of the analysis of variance showed that glycerol and maleic acid concentrations did not have any significant effects. This indicates that the ester bonds formed were not sensitive to the portion range used in this study. In other words, the smallest portions of those reagents could be used to produce GEMR.

Ash content measurement was carried out to determine the levels of foreign materials, acid insoluble materials, calcium, potassium, and magnesium salts (Mocak *et al.* 1998). Ash content indicates the amount of mineral content in a product (Waluyo *et al.* 2012). The value is important for GEMR applied to paper products. The obtained average value of ash content ranged from 5 x 10^{-3}% to 14.7 x 10^{-3}%. The results of the analysis of variance showed that the only factor that had a significant effect was the maleic acid concentration (p = 0.007). Significant differences were observed at maleic acid concentrations of 8% and 12% (Fig. 3). It is assumed that the maleic acid contained some impurities during the production of GEMR.

The solubility in toluene was measured qualitatively. The GEMR product was completely soluble in toluene at all concentrations of glycerol and maleic acid. This indicates low levels of impurities in rosin (Khadafi *et al.* 2014). Organic solvents such as ethyl ether, benzene, and ethyl alcohol are able to dissolve gum rosin (Permatasari & Rahmatullah 2018). More polar alcohol solvents would dissolve rosin faster than toluene would.

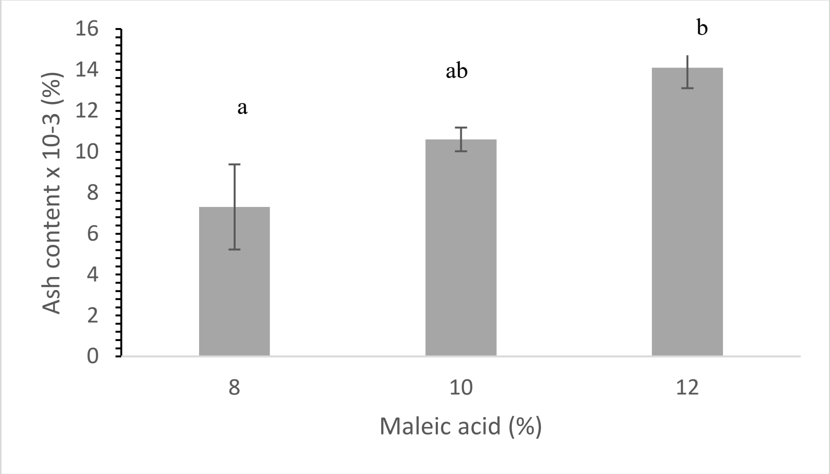


Figure 3. Effect of maleic acid percentage (based on rosin weight) on the ash content value. The same letters are not statistically different at $P < 0.05$ by Tukey's test.

Comparison of Glycerol Ester of Rosin Maleic with Control Rosin

Rosin derivatives were processed to overcome gum rosin weaknesses. These weaknesses could be reduced by gum rosin modification by methods such as esterification (Soliman *et al.* 2021) and fortification using maleic acid (Bildik *et al.* 2019). The comparison of statistics between the rosin starting material and the GEMR produced is presented in Table 3. There was no difference between both in the solubility in toluene. However, a considerable difference was observed in the acid number, in which case GEMR had significantly lower values (190.49 mg KOH/g vs 5.84~9.82 mg KOH/g). The esterification reaction could be evaluated by a decrease in the acid number (Rachmawati 2009). The acids in the rosin underwent a decrease in the number of H atoms due to binding to OH atoms during the esterification process (Kirk and Othmer 2004). On the other hand, the softening point of GEMR drastically increased compared to the rosin control (92~121ºC vs of 75.07ºC). This was because the GEMR had undergone a long cooking process at high temperatures. Thus, the turpentine amount diminished. A different trend was observed for the ash content. The GEMR showed slightly different values than those of the original rosin. The highest ash content level was 15 x 10^{-3}%, which indicates that the GEMR product contained small amounts of minerals. On the contrary, the lowest ash content value was 5 x 10^{-3}%, which suggests that some minerals were degraded during the heating process.

Table 3. Properties of glycerol ester of maleic rosin, control rosin, and gum rosin of SNI 7636:2020

	Glycerol Ester of Rosin Maleic									
Properties	Concentration of Glycerol (%)	Concentration of Maleic Acid (%)			Gum Rosin (Control)	SNI 7636:2020*				
		8	10	12						
Acid Number (mg KOH/g)	10	10.04	7.71	9.82	190.49	160~200				
	12	7.95	7.48	9.12						
	14	7.71	5.84	8.88						
Softening Point (ºC)	10	92	93	104	75.07	≥78	≥78	≥78	≥76	≥74
	12	107	121	105						
	14	105	104	113						
Ash Content (× 10^{-3} %)	10	8.7	11.3	14.7	9	≤ 20	≤ 20	≤ 40	≤ 50	≤ 80
	12	7.7	11	13.3						
	14	5.0	9.5	14.3						

Source: BSN (2020)

Comparison of Glycerol Ester of Rosin Maleic with Indonesian National Standard (SNI) for Gum Rosin

Color testing was carried out by comparing the color of the GEMR product with SNI requirements for gum rosin color. The dark brown color of GEMR (Fig. 4) was caused by lengthy heating during the fortification and esterification processes (Kencanawati *et al.* 2017). Higher maleic anhydride proportions yield a darker color (Prakoso *et al.* 2021). Thus, bleaching should be performed during the GEMR processing to obtain a clear color. With regard to the ash content, all treatments yielded GEMR products in the gum rosin range set out under the SNI (Table 3), but the softening point of the GEMR products was higher than required by the SNI. GEMR has a long carbon chain due to excessive heating during the fortification and esterification processes. The number of double bonds and the length of the carbon chain in the compounds would affect the softening point value (Ramadhiani *et al.* 2020). Furthermore, the acid number of GEMR were below the range prescribed by the SNI due to the esterification process. For food industry purposes, it is necessary to maintain the lower values of acid number (Hidayat *et al.* 2021).

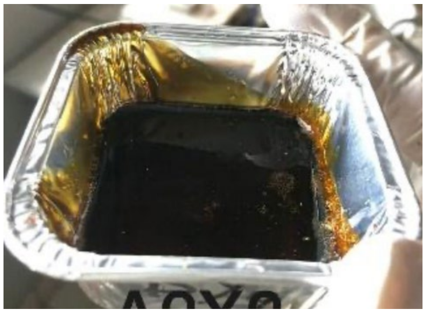


Figure 4. Glycerol ester of rosin maleic product

Comparison Glycerol Ester of Rosin Maleic with Chinese derivative gum rosin

As China has been known as the world's biggest producer of gum rosin, the research results were further compared to Chinese standards (Table 4). An acid number test was carried out to determine the quality of the gum rosin derivative product. The esterification process primarily aims to reduce the acid number or increase the rosin ester conversion using heterogeneous catalysts (Mardiah *et al.* 2023). In a previous study, Mahendra (2019) produced glycerol ester of rosin with acid number in the range from 80 to 90 mg KOH/g. The average acid number obtained in this research was in the range 5.84~9.82 mg KOH/g, lower than the value range of the four types of Chinese quality standards (GER-100M, GER-120M, GER-130M, and GER-140M).

The softening point value indicates the ripeness of the gum rosin derivative product. In this research, the softening point values of GEMR did not meet the Chinese standards in all treatments. The treatment with 14% glycerol and 8% maleic acid met the Chinese GER-100M standard, and the treatment with 12% glycerol and 10% maleic acid met the Chinese GER-120M standard. Furthermore, the solubility in toluene results indicated no undissolved materials in this experiment.

Table 4. Properties of glycerol ester of maleic rosin from the experiment and Chinese standard

Properties	Concentration of Glycerol (%)	Concentration of Maleic Acid (%)			Chinese glycerol ester of maleic rosin			
		8%	10%	12%	GER-100M	GER-120M	GER-130M	GER-140M
Acid number (mg KOH/g)	10%	8.42	7.71	9.82	≤ 25	≤30	≤ 30	≤ 42
	12%	7.95	7.48	9.12				
	14%	7.71	5.84	8.88				
Softening Point (^{o}C)	10%	92	93	104	102~108	120~126	130~136	135~145
	12%	107	121	105				
	14%	105	104	113				

Source: Wuzhou (2005)

Conclusions

From the results of this research, it can be concluded that esterification with mixes of maleic acid (at concentrations of 8%, 10%, and 12%) and glycerol (at concentrations of 10%, 12%, and 14%) had significant effects on ash content and softening points. The increase in the percentage of glycerol would result in decreases in ash content and acid number and increases in softening point (from 10% to 12% glycerol concentration). The increase in maleic acid concentration would significantly increase the ash content. No significant effect of concentrations was observed on the yield and acid number. Compared to the SNI standard for gum rosin, the GEMR produced here had lower acid number and higher softening point. It also had better quality in acid number compared to Chinese GEMR standards. Processing pine resin with the addition of maleic acid at 6% and glycerol at 10%, or with the addition of maleic acid at 6% and glycerol at 12%, fulfilled the requirement of Chinese GEMR standards for softening point.

Acknowledgments

The author thanks to KEDAIREKA program (2022) for financing this research.

References

Badan Standardisasi Nasional. 2020. Standar Nasional Indonesia 7636: Gondorukem. Jakarta.

Bildik, A. E.; M. A. Hubbe, M.E. Gule. 2019. Neutral/alkaline sizing of paper with fortified, saponified wood rosin premixed with alum and retained using cationic polymer. Appita Journal 72(1):41-51.

Djatmiko, B.; B. Sumadiwangsa, S. Ketaren. 1973. Pengujian kualitas gondorukem. Jurnal Publikasi Khusus 10(1): 4–19.

Coppen, J.J.W. G.A. Hone. 1995. Gum naval stores: Turpentine and rosin from pine resin. Food and Agriculture Organization of The United Nations: Rome.

Gomez, K. A., & Gomez, A. A. (1995). *Prosedur statistik untuk penelitian pertanian*. Universitas Indonesia: Jakarta.

Fiebach, K. 1993. Resins, Natural, dalam Ullmann's, "Encyclopedia of Industrial Chemistry, vol. A23, pp 73-88, VCH Verlagsgesellschaft, Federal Republic of Germany.

Hartanto, D. T.; Rochmadi, M.O. Wahyu, D. Kusumawati. 2021. Characteristics and kinetics study of glycerolabietate from glycerol and abietic acid from rosin. Jurnal Rekayasa Proses 15(2):170–181.

Hidayat, R.A.N.; S. Nugroho, H. Dewajani, A. Yuni. 2021. Peningkatan kualitas gondorukem dengan penambahan chelating agent dan adsorben pada proses pengolahan getah karet (*Pinus merkusii*) di PT. Perhutani Anugerah Kimia. Jurnal Teknologi Separasi 7(2):390–399. Http://Distilat.Polinema.Ac.Id

Hiller, K.; M.F. Herzig. 2007. Die große Enzyklopaedie der Arzneipflanzen und Drogen, Elsvier Spektrum, Heidelberg

Kencanawati, C. I. P. K.; I.K.G. Sugita, N.P. Gede. 2017. Characteristics and early analysis of pine resin under heating variations as alternative resins on composites. Prosiding SNTTM XVI. Oktober 2017, pp. 117-120

Khadafi, M.; I.Rostika, T. Hidayat. 2014. Pengolahan gondorukem menjadi bahan pendarihan sebagai aditif pada pembuatan kertas. Jurnal Selulosa 4(1):17–24. Http://Dx.Doi.Org/10.25269/Jsel.V4i01.53

Khadijah, A. I.; A. Chumaidi. 2022. Pengaruh volume asam klorida terhadap karakteristik fisik disproportionated rosin (DPR) dari bahan baku gum rosin tipe WG. Jurnal Teknologi Separasi 8(3):621–626. Http://Distilat.Polinema.Ac.Id

Kirk, R. E.; & D. F. Othmer. 2004. Carbon and graphite fibers to chlorocabons and chlorohydrocarbons. In: Chemical Technology (4th Ed., Vol. 5, Pp. 381–397).

Mahendra, V. 2019. Rosin product review. Applied Mechanics and Materials 890:77–91. doi:10.4028/www.scientific.net/AMM.890.77

Mardiah, M.; T. W. Samadhi, W. Wulandari, A. Aqsha, Y. A. Situmorang, A.Indarto. 2023. Recent progress on catalytic of rosin esterification using different agents of reactant. AgriEngineering 5(4):2155-2169.

Mocak, J.; P., Jurasek, G.O. Phillips, S. Varga, E. Casadei, B.N. Chikemai. 1998. The classification of natural gums. X. Chemometric characterization of exudates gums that conform to the revised specification of the gum Arabic for food use, and the identification of adulterants. Food hydrocolloids 12:141–150. https://doi.org/10.1016/S0268-005X(98)00008-3

Nurdiansyah, F. F.; Sulardjaka, N. Iskandar, 2021. Pengaruh fraksi massa dan arah orientasi serat terhadap kekuatan tegangan geser komposit berpenguat serat rami dengan matriks gondorukem. Jurnal Teknik Mesin *9*(1):81–90. Https://Ejournal3.Undip.Ac.Id/Index.Php/Jtm/Article/View/35308

Permatasari, S.; R.B. Rahmatullah. 2018. Pemisahan terpentin dan gondorukem dari getah pinus (*Pinus merkusii* Jungh. et de Vriese) dengan metode destilasi. Institusi Teknologi Sepuluh November Perum Perhutani.

Perum Perhutani. 2018. *Laporan Tahunan 2018 : Execute Now.* Jakarta : Perhutani

Prakoso, T., I. Kumalasari, B. Jiwandaru, T. Hernas , Soerawidjaja, M.M. Azis , A. Indarto. 2021. Synthesis of maleic-modified rosin ester from pine rosin. IOP Conf. Ser.: Mater. Sci. Eng. 1143 012071.doi:10.1088/1757-899X/1143/1/012071

Prasetyo, A. E.; A. Widhi, Widayat. 2012. Potensi gliserol dalam pembuatan turunan gliserol melalui proses esterifikasi. Jurnal Ilmu Lingkungan 10(1):26–31. Http://Ejournal.Undip.Ac.Id/Index.Php/Ilmulingkungan

Purnavita, S.; S. Sutanti, R.W. Sudrajat. 2017. Formulasi vernis poliester berbasis gondorukem - asam laktat dan gliserol dengan katalis $SnCl_2$. Jurnal Inovasi Teknik Kimia 2(1): 49 – 53.

Putri, D. Q. A.; A. Chumaidi. 2021. Sintesa DPR (Disproportionated rosin) dari gum rosin grade X secara batch. Jurnal Teknologi Separasi 7(2):302–309. Http://Distilat.Polinema.Ac.Id

Rachmawati, A. 2009. Sintesis Katalis Padatan Asam Γ-Al2o3 /So42- dan Digunakan pada Sintesis Senyawa Metil Ester Asam Lemak dari Limbah Produksi Margarin Minyak Kelapa Sawit. Universitas Indonesia

Ramadhiani, N.; K. Usri, O.Taofik. 2021. Perbedaan titik lunak resin damar mata kucing dengan resin damar batu. Jurnal Material Kedokteran Gigi 9(2):34 – 38. DOI: 10.32793/jmkg.v9i2.593

Rongxian, O.; W.Q. Wen. 2009. Effects of maleic rosin on the rheological properties of wood flour/HDPE composites. Scientia Silvae Sinicae 45(5):126 – 131.

Santosa, G. 2010. Pemanenan Hasil Hutan Bukan Kayu. PH Cipta Press: Papua

Soliman, A.A.; N.A. Alian, M.M. Elsawy, N.O. Shaker. 2022. Characterization and evaluation of novel sustainable polymers derived from renewable rosin. Pigment & Resin Technology 51(6):600-608.

Waluyo, T. K.; I.Wahyudi, G.Santosa, 2012. Pengaruh metode dan arah sadap terhadap produksi getah jelutung Hutan Tanaman Industri. Jurnal Penelitian Hasil Hutan 30(4):301-313.

Wibowo, G. D. H. 2013. Analisis kebijakan pengelolaan Hasil Hutan Bukan Kayu (HHBK) di NTB dan NTT. Jurnal Hukum Dan Pembangunan 43(2):180–203. Http://Dx.Doi.Org/10.21143/Jhp.

Wiyono, B. 2007. Pengaruh konsentrasi bahan kimia maleat anhidrida terhadap gondorukem maleat dari getah *Pinus merkusii*. Jurnal Penelitian Hasil Hutan 25(1):28–40.

Wuzhou. 2005. Glycerol Ester of Rosin Maleic. Shun Shine Forestry & Chemicals Ltd. Guangxi, China. http://wssfc.com/en/product-42698-50898-171202.html

Rafli Nur Ramadhani*, Ganis Lukmandaru, Sigit Sunarta
Department of Forest Products Technology, Faculty of Forestry, Universitas Gadjah Mada,
Jl. Agro No. 1, Bulaksumur, Yogyakarta, Indonesia
Tel. : +62-274-6491428 ,
Fax. : +62-274-550541
*Email : rafli.nur.ramadhani@mail.ugm.ac.id

Widiyatno
Department of Silviculture, Faculty of Forestry, Universitas Gadjah Mada,
Jl. Agro No. 1, Bulaksumur, Yogyakarta, Indonesia
Tel. : +62-274-6491428
Fax. : +62-274-550541

Antifungal Activity and Identification of Active Compounds From Wood *Tristaniopsis whiteana* (Griff) Against Wood Rot Fungus

Renhart Jemi, Wasrin Syafii, Fauzi Febrianto, Muhammad Hanafi

Abstract

The aim of the study was to analyze the content of pelawan wood extractive substances (*Tristaniopsis whiteana* (Griff)) and to test it with the fungi *Schizophyllum commune* Fr and *Pleurotus ostreatus*. Pelawan heartwood powder was macerated with methanol. Then fractionated in stages with n-hexane, chloroform, ethyl acetate, and butanol. The resulting extract was then tested with *S. commune* and *P. ostreatus* fungi. The most active fraction was isolated using column chromatography with a gradient system, the eluent was methanol:chloroform. Sub-fractions were then tested for fungi to determine the most active sub-fractions, and the most active sub-fractions were then analyzed by 1H NMR. The results showed that the extractive content of pelawan wood was most soluble in chloroform. All extract fractions contain potential as anti-fungal. .The chloroform fraction was very active compared to the other fractions. Isolation of the chloroform fraction by column chromatography obtained 8 sub-fractions. All of these sub-fractions were able to inhibit the growth of *S.commune* and *P.ostreatus* with $IC_{(50)}$ = 54.55 - 64.69 mg/L and IC(50) = 54.17 - 64.44 mg/L respectively. PL.3 sub-fraction was the most active among the 8 sub-fractions. The results of ^{1}H NMR analysis on the PL.3 subfraction were shown to be Heptanoic Acid compounds.

Keywords: Chloroform, Heptanoic acid, NMR, *Schzophyllum commune* Fr, *Pleurotus ostreatus*.

Introduction

Fungi are organisms that can cause damage to agricultural plants, forest trees, wood and processed products made from wood. Some fungi are also useful in agricultural processes, pharmaceutical industry and biotechnology such as biopulping, bioremediation, biofuel (Zabed *et al*. 2019; Ghosh, 2020, Singh *et al*. 2020, Moya *et al*. 2023). Fungi attack wood because wood is a substrate that contains lignocellulosic materials.

The lignocellulosic material in wood can be damaged by wood rotting fungi with a hydrolytic enzyme system. Wood with a low durability class is very easily attacked by wood rot fungi. The impact of wood decay fungus attack is that it can reduce the quality and aesthetics of the wood. Rotting fungi are more capable of reducing the weight of wood than termite attacks (Goodell & Nielsen, 2023). The *P. ostreatus* fungus is able to remove 27.4% of lignin and 1.58% of cellulose in Beech wood (Bari *et al*. 2021). To overcome attacks by wood rot fungi, the wood needs to be preserved before use. However, the synthetic wood preservatives used can have a negative impact on the environment and human health and these materials cannot be renewed (Meena, 2022; Changotra *et al*., 2024). Hence, it is necessary to study biodegradable natural preservatives which the ingredients are easy to obtain.

Antifungal ingredients can be obtained from wood which has natural durability, because it contains toxic extractive substances. Extractive substances are secondary metabolic compounds that have ecological functions (Vek *et al*. 2022; Mai & Zhang, 2023).

Tropical forests in Indonesia are rich in wood species that are naturally durable against wood-destroying organisms attacks. One of them is pelawan wood (*Tristaniopsis whiteana*). *T. whiteana* wood belongs to the *Myrtaceae* family. There are 40 species of *Tristaniopsis*, from shrubs to trees. This wood is used as building material, making boats, bridges and floors (Denny *et al*. 2019; Hartanto, 2019; Aldi *et al*. 2021). Pelawan wood is resistant to fungus, termite and sea worms attacks and is classified as durable class I and II, and strong class I (Muslich & Sumarni, 2005; Jasni. 2016). Several species of pelawan wood have been studied for their wood, bark and leaves, such as *T. laurina, T. obovate, T. mergu*ensi which are anti-inflammatory, anti-diabetic, antioxidant, antibacterial and anti-toxic (Enggiwanto *et al*. 2018; Al Kadri *et al*. 2019; Pratiwi, 2019; Roanisca *et al*. 2019; Mahardika *et al*. 2020; Kusuma *et al*. 2022; Mathew *et al.* 2022).

Based on the description above, it is known that the *T. whiteana* wood species has bioactive potential. However, the antifungal activity of rotted wood on *T. whiteana* wooden terraces has never been reported. This research aims to analyze and determine the content of extractive substances, antifungal activity and to identify active compounds from *T. whiteana* wood.

Materials and Methods

Making Wood Powder

Pelawan wood with a diameter of 25 cm was obtained from tropical natural forests in Hanua Ramang Village, Central Kalimantan Province. Before being used as research material, the wood was first identified at the BRIN Biological

Research Center, Cibinong, to determine the correct scientific name. The heartwood is made into powder with a size of 40 mesh (Santoso *et al*. 2020). The wood powder obtained is then air-dried naturally until it reaches air-dry conditions (moisture content 12-15%). The resulting sawdust is then put into a plastic bag and closed tightly.

Maceration of Wood Powder

The maceration process referred to a procedure by El Aziz *et al*. (2019). 2000 g of pelawan sawdust were macerated with methanol solvent at room temperature and 1 atm pressure for 48 hours. Stirring was applied during the maceration process. The ratio of sawdust to solvent was 1:3 (v/v). The maceration process was carried out repeatedly with stirring every three hours, for 15 minutes until a clear filtrate was obtained. The methanol extract of pelawan wood was evaporated using a rotary evaporator at a temperature of 40°C and a pressure of 1 atm to obtain a crude extract. The dry weight of the methanol extract was determined in the following way: 10 ml was taken from the methanol extract, then concentrated at 40°C until the extract was crystallized. After cooling,the dry weight of the methanol extract was determined. The methanol extract content was calculated based on the percentage of the solid weight of the methanol extract to the dry weight of the sawdust kiln.

Extract Fractionation

The fractionation procedure refers to the procedure of Lezoul *et al*. (2020). 100 ml of methanol extract was fractionated step by step with solvents namely n-hexane, chloroform, ethyl acetate and butanol. The crude extract mixture was shaken in a separating funnel for 10-15 minutes until the dissolved n-hexane extract and the crude extract dissolved in methanol were separated. Mixing was carried out until the filtrate becomes clear. The remaining fraction was then fractionated sequentially using chloroform, ethyl acetate, and butanol solvents. The extract filtrate of each fraction was concentrated using a rotary vacuum evaporator.

Column Chromatography Fractionation

The gravity column chromotography procedure referred to the procedure of Elfirta *et al*. (2018). The column was 50 mm in diameter and 100 cm long, containing 20 g G 60 (230 mesh) Merck silica gel. 0.5 g of the active fraction was placed in a column containing silica gel and methanol: chloroform eluent using a gradient system. .The extract obtained was collected in a 10 mL test tube. Next, analyzed using thin layer chromotography (TLC), the eluted fraction with the same Rf value was collected and evaporated using a rotary vacuum evaporator.

Cultivation of Wood Decay Fungi

The wood rot fungi used were *S. commune and P. ostreatus*, which are classified as white rot fungi (Li *et al*. 2022). The fungus was obtained from the IPB Pathology Laboratory. The fungus was first cultured on potato dextrose agar (PDA) growth medium for seven days. The composition of the growth media referred to the composition of Kusuma *et al*. (2004) as follows: 1 liter of growth medium contains 50 g of glucose, 120 g of onion extract, 50 g of glucose, 0.3 g of K2HPO4, 0.2 g of MgSO4.7H2O, 5 g of polyptone, and 30 g of agar powder, pH 6.0.

Testing of Anti-fungal Activity

The testing of all extractive substances were obtained from maceration, fractionation and column chromotography results. The treatments were variations in the concentration of extractive substances as follows: 0 mg/L, 50 mg/L, 100 mg/L, 250 mg/L, 500 mg/L and 1000 mg/L. The positive control was CCB (copper-chrome-boron) wood preservative with a concentration of 100 mg/L. Firstly, the petri dish was autoclaved for 15 minutes at a temperature of ± 120°C with a pressure of 1 atm. Filled with media and extractive substances according to the treatment, each treatment was repeated 3 times and planted with *S. commune* and *P. ostreatus* fungi. Then, it was incubated at 25°C for 7 days in a dark room. Fungal growth inhibitory activity by extractive substances was carried out at the end of the incubation period, by measuring the growth diameter and comparing it with the growth of control mycelium. The basis for determining anti-fungal activity uses the following formula Kamaruzzaman *et al*. (2021) :

$$P = \frac{C - T}{C} \times 100\%$$

Note, *T* is the area of mycelium in the treated petri dish; *C* is the area of mycelium in the control petri dish; P is the percentage of mycelium growth inhibition.

Characteristics of Compounds with a 1H NMR Spectrophotometer

The compound structure of isolated 0.5 mg of the most active extract was determined using 1H NMR. Analysis was conducted at Advanced Chemical II Characteristics Laboratory, BRIN Serpong, Tangerang.

Data Analysis

Data on the percentage of fungal growth inhibition was analyzed to determine the $IC_{(50)}$ value. Determination was based on the logarithmic equation between the concentration of pelawan wood extract (y-axis) and the percentage of inhibition of fungal growth (x-axis). Calculations using the SPSS 17.0 program.

Results and Discussion

Pelawan Wood Extract Content

Maceration with methanol solvent was able to dissolve 104.39 g of pelawan wood extract (equivalent to 6%). This happens because the solvent has polar covalent bonds which will be polarized to form a partial charge which is able to attract electrons from the compounds in the pelawan wood. The results of maceration and extractive fractionation of Pelawan heartwood are presented in Table 1. The difference in extract content in each fraction is caused by differences in the polarity of each solvent. In terms of quantity, pelawan wood extract contains more non-polar compounds. Asmah *et al.* (2020) reported that the extraction of orange peel, *Dillenia spp* wood with chloroform was able to dissolve D-Limone, fatty acids, and wax. The n-hexane solvent dissolves wax, essential oils, vegetable oils and fats (Zhuang *et al*. 2018; Naqvi *et al*. 2020), the ethyl acetate solvent is able to dissolve alkaloids, terpenoids, flavonoids and aglycones (Kamalarajan *et al*. 2020). Butanol solvent is able to dissolve phenolic compounds, flavonoids, and saponins (Cai *et al*. 2021; Nurcahyo *et al*. 2022).

Table 1**.** Content of Pelawan heartwood extract

Fraction	Solid weight (g)	Extractive (%)
Dissolved n-hexane	0.21	0.01
Dissolved chloroform	55.61	3.26
Dissolved ethyl acetate	28.45	1.64
Dissolved butanol	1.19	0.06
Not Dissolved	17.93	1.03
Methanol extract (Amount)	104.39	6.00

Antifungal Activity of Pelawan Wood Extract

To determine the percentage of growth inhibition of the test fungus, pelawan wood extract dissolved in several solvents was then tested for *S. commune* and *P. ostreatus* fungi. . The results of the fungal growth inhibition test are shown in Figure 1 and Figure 2. Figure 1 displays a graph of the tendency to inhibit the growth of the fungus *S. commune* which is almost the same from a concentration of 50-1000 mg/L in each pelawan wood extract dissolved in n-hexane, chloroform, butanol. These three extracts are able to inhibit fungal growth by more than 68-79%. It exceeds the capabilities of CCB synthetic preservatives. Meanwhile, in Figure 2, the graph shows the trend of inhibiting the growth of *P. ostreatus* fungus, which continues to increase as the extract concentration increases. Pelawan wood extract was more active in inhibiting the growth of the fungus *S. commune* than *P. ostreatus*. This difference is due to the differences in extractive compounds contained in each fraction. Apart from that, the two types of fungi produce different enzymes for food metabolism (Kumar *et al*. 2022; Ibarra-Islas, *et al*. 2023). The difference in inhibition of *S. commune* fungal growth at a concentration of 50 mg/L n-hexane fraction was 75%, higher than the positive control. Because n-hexane extract of pelawan wood is able to inhibit non-enzymes and hydrolytic enzymes released by the fungus *S. commune* (Suryadi *et al*. 2022). The growth inhibition of *P. ostreatus* at an extract concentration of 50 mg/L chloroform fraction was 53%, also higher than the positive control. Differences in fungal growth inhibitory activity in pelawan wood extract, because the fungus *S. coummune* degrades cellulose while *P. ostreatus* degrades lignin in wood. The 100 mg/L CCB control was not able to inhibit the growth of *S. commune* and *P. ostreatus* fungi optimally because CCB was able to bind to the oxalic acid produced by *S. commune* and *P. ostreatus* fungi, then CCB no longer works. (Palmieri *et al*. 2019).

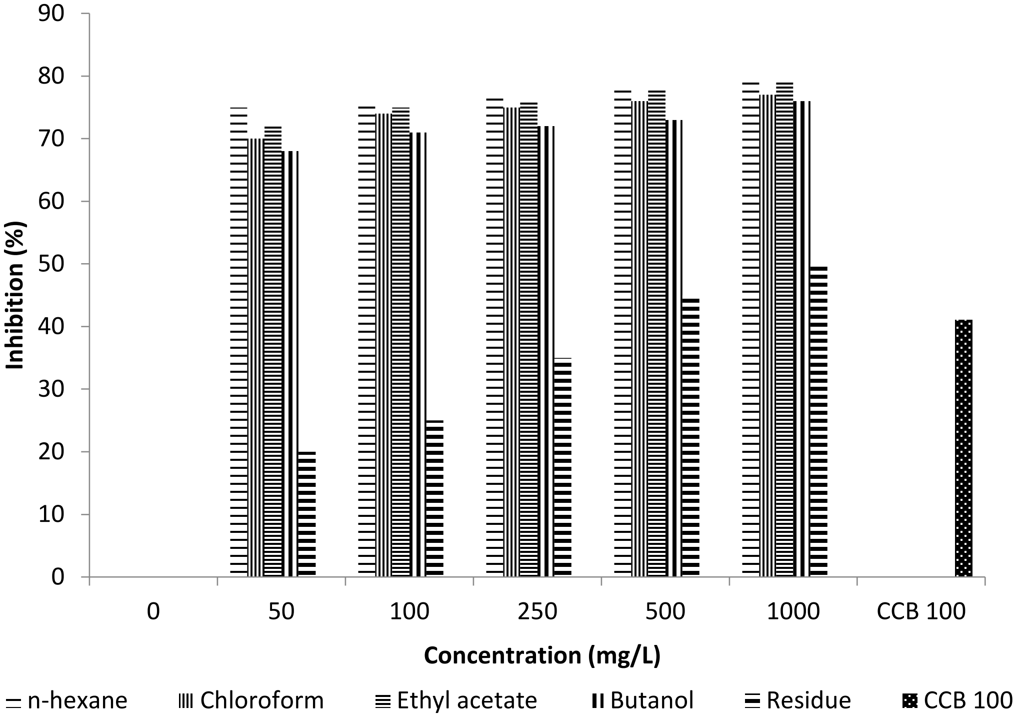


Figure 1. Inhibition of *S. Commune* mycelium growth at several concentrations of Pelawan wood extract.

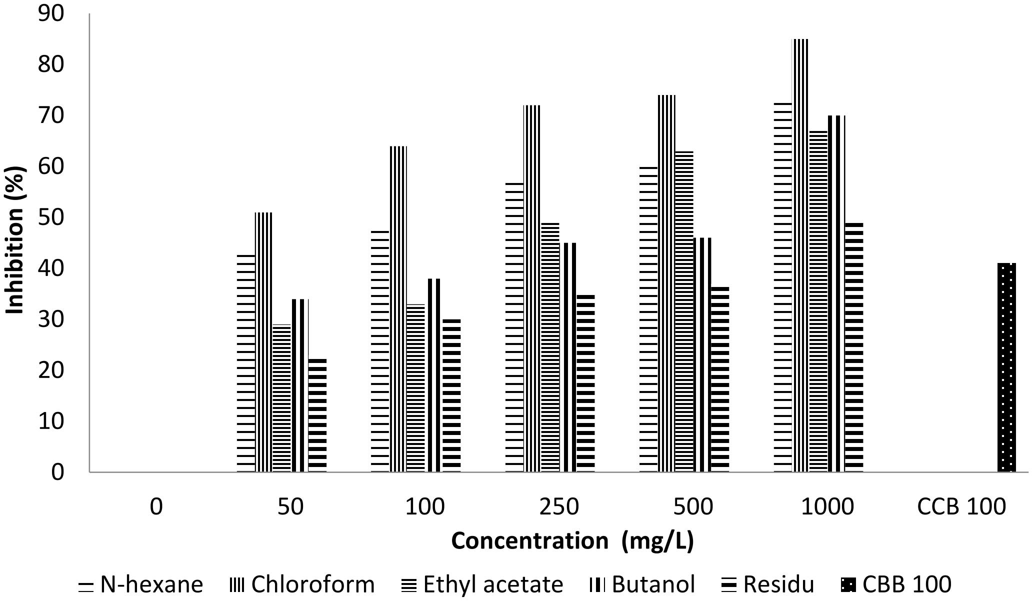


Figure 2. Inhibition of *P. ostreatus* mycelium growth at several concentrations of Pelawan wood extract

Isolation and Antifungal Activity of Chloroform Fraction from Pelawan Wood Extract

The selected pelawan heartwood chloroform fraction was isolated using column chromatography, because the results of testing the anti-fungal activity of the chloroform fraction extract were the most active of the 4 fractions. The chloroform fraction at a concentration of 50 mg/L was able to inhibit the growth of *S. commune* by 70% and *P. ostreatus* by 51%. In addition, the chloroform fraction produced a large yield of 55.61 g, making it is possible to isolate using column chromatography. A total of 0.61 g of the chloroform fraction was extracted again using column chromatography. The eluent is methanol: chloroform with a gradient system. The compounds obtained were combined based on thin layer chromatography (TLC) analysis, the results obtained for sub fraction 8 (PL.1-PL.8), in full are presented in Table 2.

Tabel 2. Eight sub-fractions of the chloroform fraction based on the results of TLC analysis

Sub faction	Weight (mg)	Rf	Value
PL.1	88.60	1.1	0.88
		1.2	0.98
PL.2	137.39	2.1	0.88
PL.3	14.50	3.1	0.73
PL.4	72.80	4.1	0.88
PL.5	10.10	5.1	0.65
		5.2	0.88
PL.6	15.10	6.1	0.85
PL.7	8.60	7.1	0.75
PL.8	18.70	8.1	0.69
		8.2	0.86

Rf = retention factor

The 8 (eight) subfractions (PL.1-PL.8) were tested for their antifungal properties to obtain the most active subfraction. The results of antifungal testing on compounds PL.1-PL.8 are shown in Figure 3. Tests on the fungi *S. commune* and *P. ostreatus* on all compounds PL.1-PL.8 showed that all subfractions were able to inhibit the growth of the fungus *S. commune* IC(50) = 54.53-64.69 mg/L and *P. ostreatus* IC(50)=54.17-64.44 mg/L. Compared to all fractions, CCB was only able to inhibit the growth of *S. commune* IC(50) = 81.49 mg/L and *P. ostreatus* IC(50) = 75.08 mg/L. This shows that the PL.1-PL.8 sub-fraction contains bioactive anti-wood rot fungal compounds whose ability exceeds that of CCB wood preservative. The PL.3 sub-fraction is the most active among the 8 sub-fractions (PL.1-PL.8), which is able to inhibit the growth of the fungus *S. commune* IC(50)= 55.09 mg/L and *P. ostreatus* IC(50)= 54.17 mg/L. Results of fungal testing on sun PL fraction. 3 is shown in full in Figure 3.

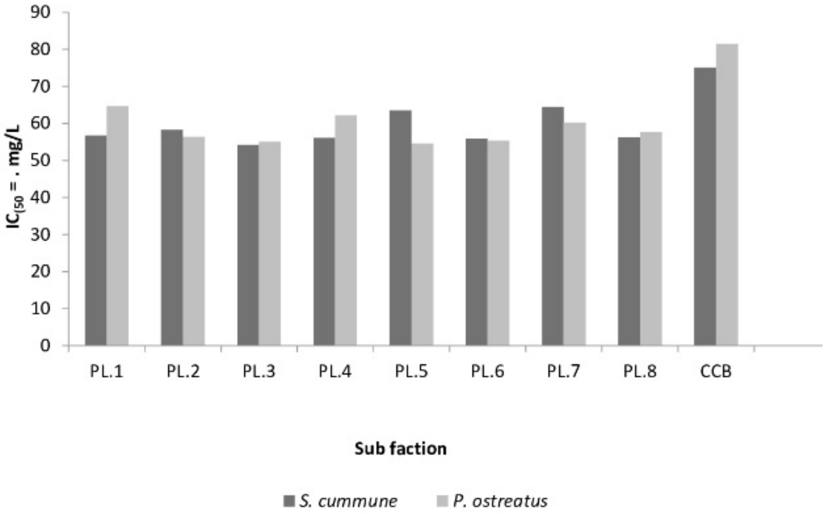


Figure 3. IC$_{(50)}$ growth of *S. commune* and *P. ostreatus* mycelium in the PL.1-PL.8 sub-fraction of Pelawan wood chloroform extract.

Identify anti-fungal compounds

The PL.3 subfraction is a thick green oil, has an unpleasant sour smell, with a boiling point: 222-245ºC. The identification results are in accordance with the reference for bioactive compounds in Buckingham (2006). The results of structural identification using 1H NMR are shown in Table 3. The structural forms of the compounds contained in the PL.3 subfraction are shown in Figure 4. As a comparison, the structure in Buckingham (2006) is used. The results of the identification of anti-fungal compounds from the PL.3 subfraction of the chloroform extract of Pelawan wood were heptanoic acid. This compound is a saturated fatty acid, with a molecular weight of 130,186 and the compound formula is C7H14O2. The 1H NMR spectrum displays signals of 7 (seven) hydrogen atoms δH 500 Mhz 1.26 (bs), 0.88 (s, J = 12.06 Hz, 3 H), the presence of a carboxylic acid group (-COOH) on C-1 and CH3 in position C-7.

Table 3. Position of 1H NMR signals for heptanoic acid

Position	δ_H 500 Mhz (ppm) (multiplicity *J* in Hz, amount H)
1	-
2 – 6	1.23 (bs)
7	0.88 (s, *J* = 12.06 Hz, 3 H)

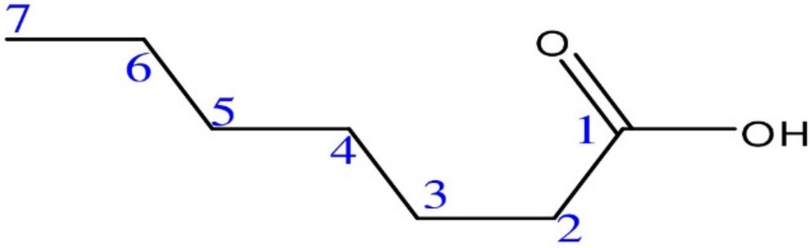


Figure 4. Heptanoic acid compound

The literature search for the mechanism of inhibition of heptanoic acid on fungal growth as follows: begins with the penetration of heptanoic acid into the cell membrane of wood rot fungi, especially at low sterol content, resulting in damage to myrysylated proteins, inhibition of the β-oxide reaction in triacylglycerol synthesis, and inhibition of topoisomerase activity (Guo *et al*. 2019; Castaño *et al.* 2022). It is because there is a carboxylic acid group in heptanoic acid which inhibits the pH regulation mechanism in cells. Finally, the function of the hydrolytic enzymes endo-1,4-β-glucoside, exo-1,4-β-glucoside and β-glucoside produced by eucalyptus rot fungal hyphae to hydrolyze cellulose into glucose is inhibited (Zhou *et al*. 2018; Sundararaj *et al.* 2020; Goodell 2020). Likewise, the enzymes O-acetyl-4-O-methyglucoroxylan and -aceylgalactoglucomannan hydrolyze Acetylarabinoglucuronomannan into glucose, mannose, galactose and acetic acid which are also inhibited by heptanoic acid (Ahuactzin-Pérez *et al*. 2018). If the penetration of heptanoic acid increases, it causes. wood rot fungi do not grow.

Conclusions

The results of multilevel fractionation showed that the chloroform fraction was the most active compared to the four n-hexane, ethyl acetate, butanol and residue fractions, was able to inhibit the growth of the fungi *S. commune* and *P. ostreatus*. The column chromatography resulted 8 subfractions (PL.1-PL8) from the chloroform fraction. The PL.3 subfraction was the most active compared to the other 7 subfractions (PL.1-PL.8) and the positive control (CCB). The PL.3 subfraction was identified as a heptanoic acid compound.

References

Ahuactzin-Pérez, M., Tlecuitl-Beristain, S., García-Dávila, J., Santacruz-Juárez, E., González-Pérez, M., Gutiérrez-Ruíz, M. C., & Sánchez, C. 2018. A novel biodegradation pathway of the endocrine-disruptor di (2-ethyl hexyl) phthalate by *Pleurotus ostreatus* based on quantum chemical investigation. *Ecotoxicology and environmental safety*, 147, 494-499. DOI: 10.1016/j.ecoenv.2017.09.004

Aldi W, Andre M.V & Aldy A. 2021. Rancang bangun mesin pembuat dowel kayu perahu nelayan. Karya Tulis. Diploma III Politeknik Manufaktur Negeri Bangka Belitung.

Anisa, Y. 2020. Sifat anti-rayap zat ekstraktif kayu pelawan (*Tristaniopsis merguensis*) terhadap rayap tanah (*Coptotermes curvignathus Holmgren*). Skripsi. Fakultas Kehutanan. Bogor Agricultural University (IPB).

Andlar, M., Rezić, T., Marđetko, N., Kracher, D., Ludwig, R., & Šantek, B. 2018. Lignocellulose degradation: An overview of fungi and fungal enzymes involved in lignocellulose degradation. *Engineering in Life Sciences*, *18*(11), 768-778. DOI; 10. 1002/eslc.201800039

Al Kadri, M. F., Sunarni, T., Pamudji, G., & Zamzani, I. 2019. Aktivitas antioksidan ekstrak etanol daun pelawan (*Tristaniopsis obovate.* Benn) dengan metode penangkapan radikal bebas 2, 2'-Difenil-1-Pikrilhidrazil. *JCPS (Journal of Current Pharmaceutical Sciences)*, *2*(2), 167-172.

Asmah, N., Suniarti, D. F., Margono, A., Mas'ud, Z. A., & Bachtiar, E. W. 2020. Identification of active compounds in ethyl acetate, chloroform, and N-hexane extracts from peels of Citrus aurantifolia from Maribaya, West Java, Indonesia. *Journal of Advanced Pharmaceutical Technology & Research*, *11*(3), 107. DOI: 10.4103/japtr.JAPTR_177_19

Bari E, Ohno K, Yilgor N, Singh AP, Morrell JJ, Pizzi A, Ghanbary MAT, Ribera J. 2021. Characterizing fungal decay of beech wood: potential for biotechnological applications. *Microorganisms*, *9*(2):247. DOI: 10.3390/microorganisms9020247

Buckingham J. 2006. *Dictionary of Natural Product.* Chapter and Hall/CRC. HDS Sofware. Hamden Data Service Ltd

Cai, X., Xiao, M., Zou, X., Tang, J., Huang, B., & Xue, H. 2021. Extraction and separation of flavonoids from *Malus hupehensis* using high-speed countercurrent chromatography based on deep eutectic solvent. *Journal of Chromatography A*, *1641*, 461998.. DOI: 10.1016/j.chroma.2021.461998

Castaño, J. D., Muñoz-Muñoz, N., Kim, Y. M., Liu, J., Yang, L., & Schilling, J. S. 2022. metabolomics highlights different life history strategies of white and brown rot wood-degrading fungi. *Msphere*, *7*(6), e00545-22. DOI: 10.1128/msphere.00545-22

Changotra, R., Rajput, H., Liu, B., & Murray, G. (2024). Occurrence, fate, and potential impacts of wood preservatives in the environment: Challenges and environmentally friendly solutions. *Chemosphere*, 141291. https://doi.org/10.1016/j.chemosphere.2024.141291

Denny, Wardani M, Kalima T, Susilo A. 2019. Pohon Hutan Rawa Gambut. Penerbit IPB Press. Kota Bogor. Jawa Barat. Indonesia.

Derba-Maceluch, M., Mitra, M., Hedenström, M., Liu, X., Gandla, M. L., Barbut, F. R., Abreu. N. A., Donev N. E., Urbancsok J, Moritz T, Janson L. J, Tsang A, Powlowsk J, Master E. R, & Mellerowicz, E. J. 2023. Xylan glucuronic acid side chains fix suberin-like aliphatic compounds to wood cell walls. *New Phytologist*. 238:297-312 DOI https://doi.org/10.1111/nph.18712

El Aziz, M. M. A., Ashour, A. S., & Melad, A. G. 2019. A review on saponins from medicinal plants: chemistry, isolation, and determination. *J. Nanomed. Res*, *8*(1), 282-288. DOI: 10.15406/jnmr.2019.07.00199

Enggiwanto, S., Istiqomah, F., Daniati, K., Roanisca, O., & Mahardika, R. G. 2018. Ekstraksi daun pelawan (*Tristaniopsis merguensis*) sebagai antioksidan menggunakan microwave assisted extraction (MAE). *Indonesian Journal of Pure and Applied Chemistry*, 1(2), 50-55.

Grąz, M., Ruminowicz-Stefaniuk, M., & Jarosz-Wilkołazka, A. 2023. Oxalic acid degradation in wood-rotting fungi. Searching for a new source of oxalate oxidase. *World Journal of Microbiology and Biotechnology*, *39*(1), 13. DOI https://doi.org/10.1007/s11274-022-03449-4

Ghosh, P.,Ghosh, U. 2020. Microbial Laccase: A vanguard biocatalyst and its potentiality towards industrial applications. *Microbial Fermentation and Enzyme Technology*, 269-282.

Goodell, B. 2020. Fungi involved in the biodeterioration and bioconversion of lignocellulose substrates. *Genetics and Biotechnology*, 369-397. DOI https://doi.org/10.1007/978-3-030-49924-2_15

Goodell, B., & Nielsen, G. 2023. Wood biodeterioration. In Springer Handbook of Wood Science and Technology (pp. 139-177). Cham: Springer International Publishing.

Guo, H., Qin, X., Wu, Y., Yu, W., Liu, J., Xi, Y., Dou., G., Wang., L.,. & Xiao, H. 2019. Biocontrol of gray mold of cherry tomatoes with the volatile organic monomer from Hanseniaspora uvarum, trans-cinnamaldehyde. *Food and Bioprocess Technology*, *12*, 1809-1820. DOI https://doi.org/10.1007/s11947-019-02319-6

Hartanto, S. 2019. Etnomedisin Tumbuhan Pelawan (*Tristaniopsis* spp.) dalam Kehidupan Masyarakat Lom Pulau Bangka. (Doctoral dissertation). Bogor Agricultural University (IPB).

Ibarra-Islas, A., Hernández, J. E. M., Armenta, S., López, J. E., López, P. M. G., León, S. H., & Arce-Cervantes, O. 2023. Use of nutshells wastes in the production of lignocellulolytic enzymes by white-rot fungi. *Brazilian Archives of Biology and Technology*, *66*. https://doi.org/10.1590/1678-4324-2023210654

Jasni. 2016. Natural durability of 57 Indonesian wood species tested under the shade. *Jurnal Penelitian Hasil Hutan*, *34*(3), 179-188.

Kamalarajan, P., Muthuraman, S., Ganesh, M. R., & Valan, M. F. 2020. Phytochemical investigation of nilavembu kudineer chooranam ethyl acetate extract and its ability to reduce intracellular antioxidant levels in THP-I cells. *European Journal of Medicinal Plants*, *30*(4), 1-13. DOI: 10.9734/ejmp/2019/v30i430187

Kamaruzzaman, M., Islam, M. S., Mahmud, S., Polash, S. A., Sultana, R., Hasan, M. A., Wang, C & Jiang, C. 2021. In vitro and in silico approach of fungal growth inhibition by *Trichoderma asperellum* HbGT6-07 derived volatile organic compounds. *Arabian Journal of Chemistry*, *14*(9), 103290. DOI:10.1016/j.arabjc.2021.103290

Kumar, A., Bharti, A. K., & Bezie, Y. 2022. *Schizophyllum commune*: A fungal cell-factory for production of valuable metabolites and enzymes. *BioResources*, *17*(3), 5420-5436. *DOI: 10.15376/biores.17.3.Kumar*

Kusuma, G. F., Mahardika, R. G., & Sari, F. I. P. 2022. Ekstrak batang pelawan (*Tristaniopsis merguensis* Griff.) sebagai antibakteri pada *Staphylococcus aureus* dan *Escherichia coli*. Stannum*: Jurnal Sains dan Terapan Kimia*, *4*(2), 40-46. DOi: 10.33019/jstk.v4i2.3063

Lezoul, N. E. H., Belkadi, M., Habibi, F., & Guillén, F. 2020. Extraction processes with several solvents on total bioactive compounds in different organs of three medicinal plants. *Molecules*, *25*(20), 4672. DOI: 10.3390/molecules25204672

Li, T., Cui, L., Song, X., Cui, X., Wei, Y., Tang, L., & Xu, Z. 2022. Wood decay fungi: An analysis of worldwide research. *Journal of Soils and Sediments*, *22*(6), 1688-1702. DOI: https://doi.org/10.1007/s11368-022-03225-9

Meena, R. K. (2022). Hazardous effect of chemical wood preservatives on environmental conditions, ecological biodiversity and human being and its alternatives through different botanicals: A review. Environment and Ecology 40 (3) : 1137—1143.

Muslich, M., & Sumarni, G. (2005). Kelas keawetan 200 jenis kayu indonesia terhadap penggerek di laut. *Jurnal Penelitian Hasil Hutan Bogor*, *4*(2), 46-49.

Naqvi, S. F., Khan, I. H., & Javaid, A. 2020. Hexane soluble bioactive components of *Chenopodium murale* stem. *Pakistan Journal of Weed Science Research*, *26*(4), 425. DOI: 10.28941/pjwsr.v26i4.875

Nurcahyo, H., Sumiwi, S. A., Halimah, E., & Wilar, G. 2022. Secondary metabolite determination from Brebes shallot's ethanol extract and its ethyl acetate fraction "*Allium ascalonicum* L.". *Pharm. Educ. Res*, *12*, 71. DOI: 10.51847/NfNMFJB9ac

Renhart Jemi
Departement of Forestry, Faculty of Agriculture, Universitas Palangka Raya, Kampus Tunjung Nyaho Jalan Yos Sudarso Palangka Raya 73111A,
Kaliamantan Tengah , Indonesia
E-mail : jemi@for.upr.ac.id

Volume 15, Number 1, April 2024 ISSN 2087-3840

WOOD RESEARCH Journal

Journal of Indonesian Wood Research Society

Annals of the Wood Research Journal

Wood Research Journal is the official journal of the Indonesian Wood Research Society. This journal is an international medium in exchanging, sharing and discussing the science and technology of wood.

Aims and Scope

The journal publishes original manuscripts of basic and applied research of wood science and technology related to Anatomy, Properties, Quality Enhancement, Machining, Engineering and Constructions, Panel and Composites, Entomology and Preservation, Chemistry, Non Wood Forest Products, Pulp and Papers, Biomass Energy, and Biotechnology. Besides that, this journal also publishes review manuscripts which topics are decided by the Editors.

Imprint

WRJ is published by Indonesian Wood Research Society
ISSN print: 2087-3840
Electronic edition is available at:
http://ejournalmapeki.org/index.php/wrj

Publication Frequency

Journal is published in one volume of two issues per year (April and October).

Peer Review Policy

WRJ reviewing policies are: Every submitted paper will be reviewed by at least two peer reviewers. Reviewing process will consider novelty, objectivity, method, scientific impact, conclusion and references.

General Remarks

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.

Editorial Address

Faculty of Forestry, Gadjah Mada University
Jl. Agrobulaksumur, Yogyakarta 55281, Indonesia

Tel/Fax : (0274) 512102, 6491420 / (0274) 550541
E-mail : fkt@ugm.ac.id; fkt-ugm@indo.net.id
Web-site : www.ejournalmapeki.org

Volume 15, Number 1, April 2024 ISSN 2087-3840

WOOD RESEARCH Journal

Journal of Indonesian Wood Research Society

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.

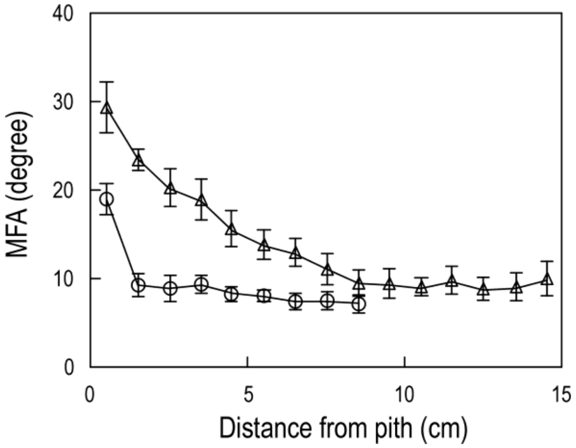


Figure 3. Radial variation of microfibril angle of the S2 layer in tracheid. Open circle, *Agathis* sp.; open triangle, *Pinus insularis*; Bars indicate the standard deviation. (Source: Ishiguri *et al.* 2010)

9 772087 384007