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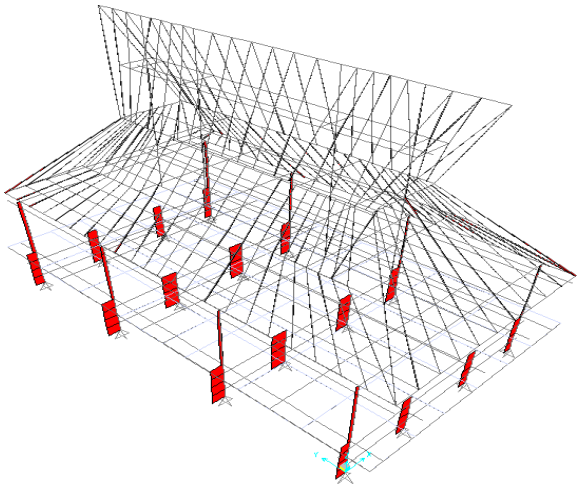

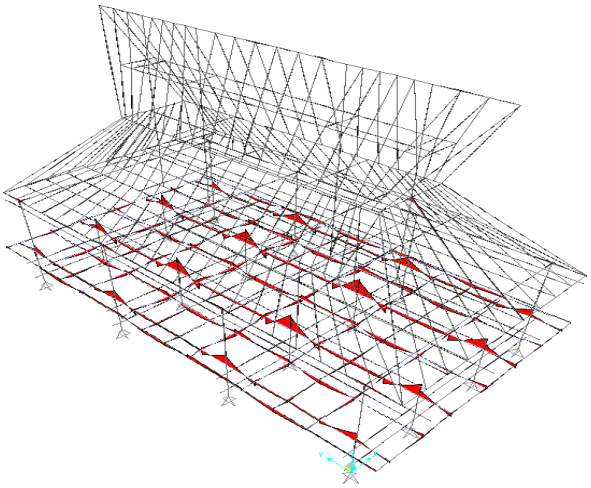

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Resisting Timber Joint Performance of Karo Wooden Building

Khairussa'diah and Yulianto P. Prihatmaji

Abstract

Batak Karo is one of the ethnic group that exists in North Sumatra. Wooden house of Batak Karo called with Siwaluh Jabu. This wooden house has a structure system of pillars on top of stone foundations. This type of foundation is able to improve the performance of the overall structure due to lateral style caused by the earthquake. This research was conducted to know the behaviour of the structure of a wooden Batak Karo home especially the restoration of wooden style joints by comparing the results of a laboratory test, numerical analysis and analysis with SAP2000. Experimental testing in the lab do the test objects as much as 3 pieces. Testing by giving a cyclic load with a capacity of 10 tons in each of the test object to damaged object. Then conduct an evaluation the behavior of the structure consist of failure modes and moment-rotational angle relationship. After the experimental test was completed, the analysis continued with validate test results with laboratory of numerical analysis. Then conducted an analysis of the power structure by using SAP2000 program to know the power of elements against the maximum tension. The analysis conducted on the overall structure of the system and the structure of mast above the foundation stone. The program was conducted with input data: the work load, etc. As the output from these programs is the element force, etc. The final results of this program are the weaknesses and advantages of structural system observed from wooden Batak Karo.

Keywords: Karo wooden structure, siwaluh jabu, timber joint, resisting performance, restoration.

Introduction

Karo tribe is one of the tribes living in the Highlands Karo, North Sumatra, Indonesia. Karo tribe has been one of district names where they lived (Karo highlands) is Karo. This tribe has their own language called Bahasa Karo and Karo script. Karo considered as part of tribal kinship Batak Karo tribe but lot of people assume that they were not part of a kinship Batak, but Karo is an independent tribe.

Wooden house of Karo known as Siwaluh Jabu. Nowadays its existence has been difficult to found. In 2011 Lingga village only four houses left, but due the earthquake that occurred a few years ago, two of them are collapsed and could not be occupied again (Prihatmaji and Widodo 2015). Siwaluh Jabu is very famous with the beauty of architecture which is typical, stout and sturdy and decorated with philosophical values ornaments. Form, function and meaning from Siwaluh Jabu illustrate the close relationship between human each other and also with natural environment. Selection of materials for Siwaluh Jabu and the construction process without the use of nails, iron or wire binding, but using pegs and rope fibers adds to the uniqueness of Siwaluh Jabu (Sembiring 2010).

Based on the results of survey we know that the main structure of this wooden house using several types of wood which are dustpan, ingul and icap wood. Siwaluh Jabu made based on knowledge from generation to generation. Although the ancient times there has been no theory of the building structure, our ancestors can create the structure that does not collapse during an earthquake. For example Omo Hada house located in Nias, North Sumatra, Rumah Gorga in Toba, North Sumatera, Tongkonan in Toraja, South Sulawesi and Uma Lengge in Mbawa, Nusa Tenggara Barat. All of those Indonesian wooden wooden house using pedestals/ rock as a foundation, pillars only placed on pedestals/ stones without using a special joint, it has been proven (Nurdiah 2011), Omo Hada wooden home when the 8.7 scale Richter Nias earthquake (2005) did not run into structural collapse (Pranata and William 2013; Pudjisuryadi *et al*. 2007).

Pillars resting on a stone serves as base isolation in a wooden house. This system is called base isolation because the poles are not contact with the ground directly, but rests on a rock. As a result, these wooden houses can move from its original location when receive the lateral seismic loads. Moreover, it can cause vibration damping effect of the earthquake (Pudjisuryadi *et al*. 2007).

Figure 1. Wooden house Siwaluh Jabu of Karo Batak tribe (Prihatmaji and Widodo 2015).

The purpose of this study is to discuss the behavior of the structural system of wooden wooden house Batak Karo due to gravity load and lateral load. Building structures and wooden material data become components of columns, beams, roof and floor boards drawn from the survey results directly.

Gravity load includes the weight of its own buildings, dead load and live load. Lateral load is based on seismic Indonesian earthquake rules of ISO 1726-2012 where for North Sumatra, the type of soil is assumed to normal type of soil which had risk buildings category IV and earthquake primary factor (Ie) 1.5 (ISO 1726-2012). Structure analysis of lateral loads using software SAP2000. Recording data is used seismic record of North Sumatra earthquake which intensity scaled to the maximum amplitude of ground acceleration (Ao) (ANSS 2015) on spectrum response curve of ISO 03-1726-2002 when T = 0.

Materials and Methods

Orthotropis Properties of Wood

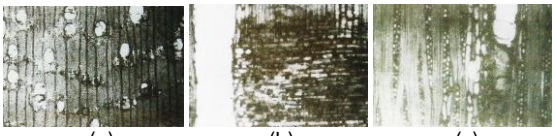
Wood has three axes of symmetry which intersect perpendicular due to the composition of the wood so that often referred to orthotropis properties. The third axis of symmetry is "longitudinal axis (extending the fiber)", "radial axis (perpendicular to growing circle)" and "tangential axis (alluded to growing circle)" (Mardikanto *et al*. 2015).

These three axis influenced by the orientation of the fiber structure, the cell radius (ray cell) as well as other wood-forming element (cell fibers, trakeida cells, parenchymal cells). The amount of wood stiffness and elasticity properties is different depending on the direction of the axis. In general, difference of these properties is determined by the fiber longitudinal direction (axial) and the perpendicular fibers (transverse) (Mardikanto *et al*. 2015). Difference in the properties of radial and tangential direction actually exist, but this difference usually very small and often overlooked.

Mechanical Properties of Wood

Material property data for mechanical properties of wood, using type of wood used Ulin (*Eusideroxylon zwageri*) with a specific gravity is 1.04 (PTHH 2005). In the properties of wood mechanical properties database, namely Atlas Wood Indonesia (PTHH 2005) of compressive strength parallel to Ulin amounted to 71.96 MPa, while the compressive strength perpendicular has been no reference. Ulin wood bending strength is equal to 109.12 MPa (proportional limit load) and amounted to 140.29 MPa (ultimate limit load / broken) (PTHH 2005). Ulin tensile strength is of 2.62 MPa (radial) and amounted to 6.19 MPa (tangential) (PTHH 2005).

Figure 2. Ulin wood (*Eusideroxylon Zwageri* T. Et. B) (PTHH 2005)



(a) (b) (c)

Figure 3. Transverse surface (a), radial surface (b) and tangesial surfae (c) of Ulin wood (PTHH 2005).

Historical Events of North Sumatra Quake

Historical events of North Sumatra quake can be seen in Table 1 below.

Table 1. Historical earthquakes North Sumatra.

Year	Latitude	Longitude	Depth	Magnitude
1972	3.274	98.522	124.00	5.30
1974	2.829	98.975	33.00	5.00
1976	3.166	99.015	180.00	5.60
1989	2.845	99.127	187.30	5.20
1990	3.322	98.401	144.90	5.10
1996	3.445	97.943	33.00	6.30
2001	3.718	97.794	139.10	5.10
2005	2.836	98.758	30.00	5.20
2006	3.390	99.079	204.00	6.30
2009	2.800	99.086	174.80	5.10
2014	2.835	99.071	170.89	5.60

Source: ANSS Composite Catalog Search (ANSS 2015).

Earthquake loading used in SAP2000 analysis using equivalent static seismic based UBC97. Because this rule become the basis of SNI 03-1726-2002 so that almost all of the input parameters are same so that the work of modeling the structure of Karo wooden house will be much easier.

Analysis of the Structural Strength from the Program SAP2000

SAP2000 program will help analyze the strength of the structural elements to the maximum voltage that occurs (Satyarno *et al*. 2012), so that it can be known weaknesses and strengths of the system structure of wooden Batak Karo house.

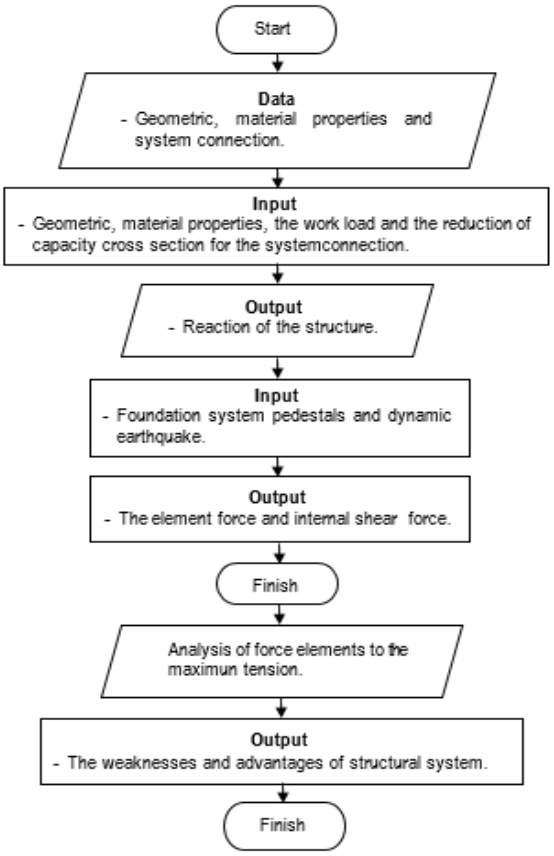


Figure 4. Analysis of the structural strength from the program SAP2000.

Results and Discussion

Structures Data and Modelling of Structures

The complete structure data taken from result of field survey on May-June 2015. The main column using cross-sectional shape of a circle with a diameter of 340 mm, the supporting pillar diameter of 300 mm. Main beam from wood with a cross section size 100x150 mm and 100x120 mm. Trunks for roof frame with a size of 140x150 mm and 90x100 mm as shown in Table 2 below.

Table 2. Data structure Karo wooden home.

House	Main pillar (mm)	Supporting pillar (mm)	Main beams (mm)	Cross-section roof (mm)
Batak Karo	Φ340	Φ300	100x150 100x120	140x150 90x100

Wood material property data used in the modeling of the structure has been described previously in the Reader Review. The results of modeling the structure shown in Fig. 5.

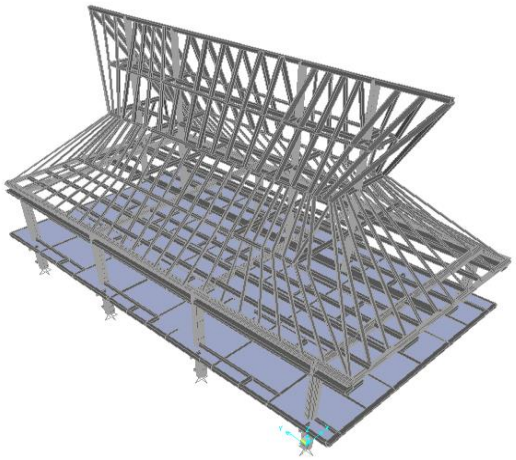


Figure 5. Result of 3D structure modeling for wooden house Karo.

In this study, the live load used is equal to 65 kg/m^2 and dead load at 6.5 kg/m^2. Earthquake loads used are seismic scaled load intensity of the maximum amplitude of ground acceleration (A_o) on spectrum response curve ISO 1726-2012 when T = 0 is equal to 0.28.

Combination of loading that used are:

(a) 1.4 DL
(b) 1.2 DL + 1.6 LL
(c) 1,3042 DL + 0.5 LL ± E

where DL is dead loads, LL is the live load, and E is the earthquake load.

The simulation results from the software SAP2000 more shown in Fig. 6 is a deformation pattern of the structure due to simulated earthquake load, and Fig. 7 (normal force/ axial happened to the pole) and Fig. 8 (bending moments that occur in the beam) where the result is taken as a result of the maximum load combinations.

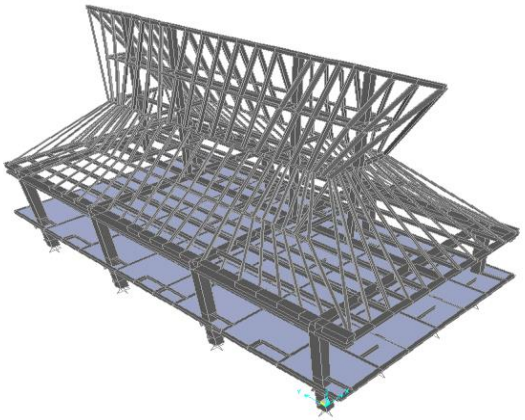
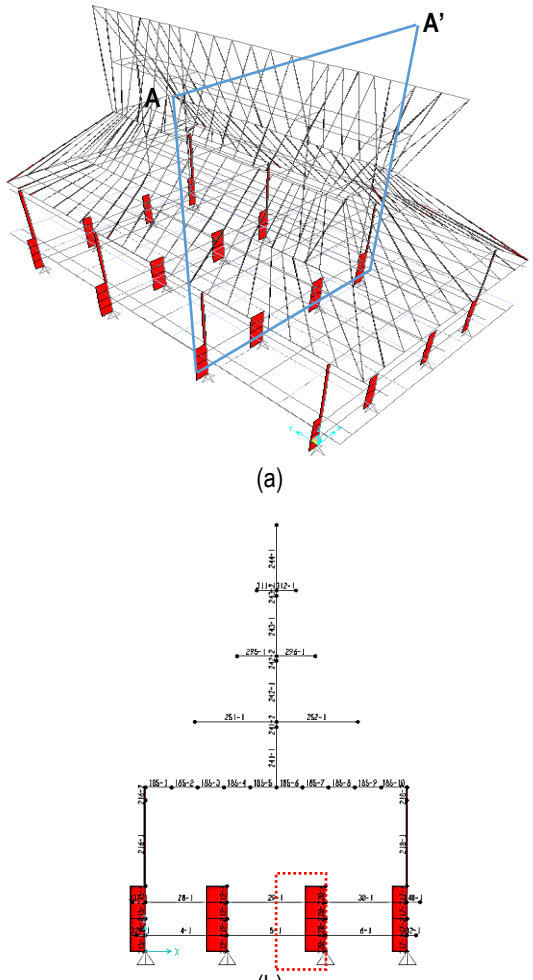


Figure 6. The pattern deformation simulation model of the structure due to earthquake load.

From Fig. 6, it can be seen that the simulation show the deformation that occurs in all poles still comply with the limits factored load combinations and load intellectually based ISO 1726-2012, so that the structure still meet the security criteria.



(a)

(b)

Figure 7. Axsonometry drawing (a), and section A-A' drawing.

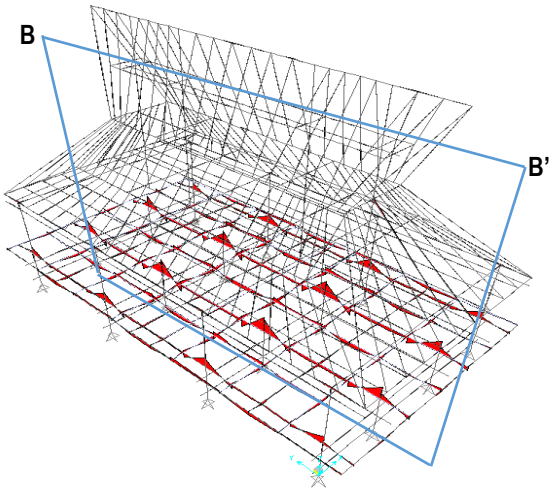
The axial forces that occur in the column is N, then the compressive strength in the column can be calculated by Equation (1) below.

$$\sigma_C = \frac{P}{A} \quad (1)$$

where σ_C is the compressive strength of the column, P is the axial force in the column, Aa is the column cross-sectional.

$$\sigma_c = \frac{P}{A} = \frac{631219{,}05}{\frac{1}{4}\times\pi\times340^2} = 6{,}952 \text{ MPa} < F_c = 71{,}96 \text{ MPa}$$

From Fig. 7, it can be seen that the simulation shows that compressive strength occurs in the column is still smaller than the compressive strength of wood is Fc of 65.24 MPa, so that the column is still in robust condition.



(a)

(b)

Figure 8. Axsonometry drawing (a), and section B-B' drawing.

The amount of bending strength that occurs can be calculated using Equation (2) below.

$$\sigma_b = \frac{M}{I} \quad (2)$$

where σ_b is the beam flexural strength, M is the bending moment on the beam, y is the distance from the beam weight to the outer edge of the fiber, and I is inertia moment of the beam.

$$\sigma_b = \frac{M}{I} = \frac{212041624}{\frac{1}{12}\times170\times65^2} = 54{,}502 \text{ MPa} < F_b = 109{,}19 \text{ MPa}$$

From Fig. 8, it can be seen that the magnitude of the flexural strength that occurs is still not exceed the limits Fb flexural strength of 109.19 MPa. Recapitulation of Batak Karo structural analysis can be seen in Table 3 and Fig. 9.

Table 3. Recapitulation of Batak Karo structural analysis.

Structural Analysis	Structural stress analysis of SAP2000 (MPa)	Allowable stress (MPa)	Explanation
Axial Force	6,952	71,96	Safe
Flexural strength	54,502	109,12	Safe

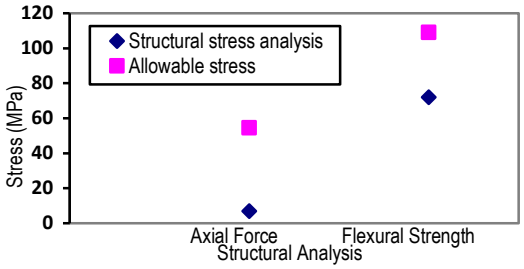


Figure 9. Structural analysis of Batak Karo house.

Conclusions

From this research, some conclusions can be drawn as follows:

The modeling results show that the deformation that in all poles still suitable for restrictions factored load combinations and load regulation based on earthquake in Indonesia. The results of the calculations show that the compressive strength of the tension that occurs at the pole is still samller than the compressive strength of wood (Fc) so that the mast is still in strong condition.

The results show that the bending strength calculation of the tension occurs in both of beam is still less than the bending strength of wood (Fb) so that the beam still in strong condition. The foundation system in Karo wooden house using stones to hold the ground shaking. It is the form of the joints, allowing the pole can hold the force due to earthquake loads and lateral loads. In general it can be concluded that due to the earthquake load and lateral load custom the structure of Karo House is in a safe condition.

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Beam-Column Timber Joint Connection Behavior Due to Nail and Modified-Washer Reinforcement Tests

Yosafat Aji Pranata, Anang Kristianto, and Olga Catherina Pattipawaej

Abstract

Timber connection capacity, in case of beam-column joint connection provides significant impact on the wooden building structures. Strength and stiffness of timber connections using reinforcement technique of wooden building structures have not been studied intensively. This paper studies the use of nails and modified-washer to improve wood connection's performance. The experimental tests were conducted in the laboratory by comparing the partial connection between test specimen timber without reinforcement (standard type) and the reinforcement (PRP type). The testing was conducted based on partial connection beam-column joint test using Universal Testing Machine's with a modified holder. Wood studied includes Meranti (*Shorea* spp.) and Mersawa (*Anisoptera* spp.). PRP type connection was using nails and modified-washer strengthening, and standard type connection was using a classic washer. Parameters studied were strength and stiffness of the connection, reviewed both: proportional limit load and ultimate limit load conditions. Result obtained from this research indicates that the use of nails and modified-washer make a positive contribution to improving the performance of the beam-column timber joint connections, in terms of strength capacity (both of proportional limit and ultimate limit loads) and stiffness capacity (displacement ductility ratio). Meranti beam-column timber joint is more brittle than Mersawa beam column timber joint, it has an impact on the results. PRP-type of Mersawa timber connection produces a higher ductility than the standard type, while the PRP-type of Meranti timber connection produces a similar ductility to the standard type.

Keywords: Partial test, beam-column joint, timber, nail and modified-washer, behavior.

Introduction

Connection performance of wooden house building structure plays an important role with regard to the overall performance of the building structure. Ductile connection systems are expected to contribute in the behavior of the strength and stiffness of the building structure positively. Fig. 1 and Fig. 2 show the beam-column joint connection in a traditional house of Minahasa, North Sulawesi, Indonesia. The observed connection is in the exterior residence (Fig. 1) and in the interior residence (Fig. 2). The wood joints were connected using nails.

Figure 1. Beam-column joint connection of Minahasa traditional house: Exterior joint.

In order to review the connection system performance, it is necessary to limit the burden of disproportionate amount of information that could be retained by connection. It is helpful in designing the timber connection to calculate lateral resistance (Z) in accordance to Indonesian National Standard (SNI 7973: 2013) (Badan Standardisasi Nasional 2013).

Figure 2. Beam-column joint connection of Minahasa traditional house: Interior joint.

Information on the load-slip curve relationship of timber connection, moment-curvature curve timber connection and ideal model approachs are also an important empirical data in relation to the numerical modeling of the wood building structure. The accuracy of numerical modeling relies heavily on modeling parameters

or idealization of the connection structure elements. The mechanical properties of the material parameters and of the cross section dimensions size of structural elements.

This study is a continuation of previous research development reported by Pranata *et al*. (2014) who mentioned that there is related research capacity of the axial tensile connections of standard type and of the nail and modified-washer reinforcements, as well as research capacity of the beam-column joint connection (Pranata *et al*. 2015). The study of standard type connection and strengthened connections using the reference of ASTM test methods (ASTM 2000).

The testing specimen concept used in this study differs from previous studies particularly in a model specimen partial connection (Pranata *et al*. 2015). In this study, the nails and modified-washer was used to improve performance of timber connections. Experimental tests i the lab were conducted by comparing partial connection between test specimens timber without reinforcement (standard type) and the reinforced specimens (PRP type). Partial beam-column joint connection test was conducted to test Meranti (*Shorea* spp.) and Mersawa (*Anisoptera* spp.) wood using Universal Testing Machine (UTM). PRP types were counducted using nails and modified-washer strengthener. Parameters studied were the strength and stiffness connection and proportional limit of loading as well as ultimate limit loading conditions. The testing method used is the monotonic loading pattern.

Due to the limited length of timber that is in-trade, then for a long timber construction timber is needed for the connection of two wooden trunks or more mutually connected to one another so that a single piece of wood long. Understanding the relationship is two sticks of wood or more interconnected with each other at a certain point that it becomes a part of the construction. Please note the terms of wooden ties, among others: as simple as possible but sturdy, attractive avoid deep wood, placement of connection, will withstand the forces acting on it. Mechanical connection can be used, among others tools connecting bolts or nails.

Basic Theory

Kobel (2011) studied the effect of strengthening, especially for connections that resist lateral loads (hereinafter referred pull axial connection) for a long-span truss. There are four types of reinforcement are studied, namely the retrofitting of type A2 + B2, strengthening 02 + A2, inclined reinforcement and Dywidag strengthening. Retrofitting is done by adding a dowel in the direction intersecting with the mechanical connection.

Noguchi *et al*. (2006) also studied the timber connection (beam-column connections), as well as developing new connection models to bolster the per-formance of the strength of the beam-column connections.

The thickness of the ring having an impact as well as the influence of pretension effect of the bolts. Pretension effect thus will not increase the capacity of joint significantly, however, a positive effect is to improving connection's ductility. Another effect is by the initials pretension then bolt becomes more difficult to bend or fail flexibly, so that it is suitable to be applied to high quality of wood with high bearing strength. Pretension with a note that the amount does not exceed the compressive strength perpendicular to the wood fibers (Awaludin *et al*. 2008a; Awaludin *et al*. 2008b).

One indicator to know stiffness is displacement ductility ratio, which is calculated by the Equation 1,

$$\mu = D_u / D_y \quad (1)$$

where μ is displacement ductility ratio, D_u is deformation due to ultimate limit load, and D_y is deformation due to proportional limit load.

Methods

The study was divided into four main stages. The first stage was the study of literature. The second stage is to study secondary data and preparation of test specimens. The third stage is the experimental testing in the laboratory. The fourth stage is processing the data to get the results of the discussion and conclusions.

The research method uses empirical methods, namely experimental testing in the laboratory. The total number of test specimens are 6 (six) specimens, which are 2 (two) specimens for Meranti timbers and 4 (four) specimens for Mersawa timbers.

Fig. 3 shows a schematic model of partial connection test object for laboratory test. Fig. 4 shows the partial connection standard type of timber connection. Fig. 5 shows the partial connection for the connection with the reinforcement (named PRP type).

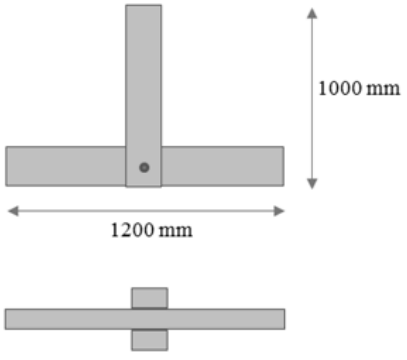

Figure 3. Schematic model of the specimen.

Specimens that used in this research are tested using a Universal Testing Machine (UTM). UTM used to apply a monotonic loads from zero load to the specimen failure. For this purpose it would require additional equipment in the

form of a holder for placement of the test object. Setups of the test specimen are shown in Fig. 6.

Figure 4. Example for the standard type of the Beam-Column Timber Joint Connection.

Figure 5. Example for the PRP type of the Beam-Column Timber Joint Connection.

Figure 6. UTM and setup of the specimen.

Results and Discussion

Testing is done by applying a load, from zero loading to the test specimen failure and could not withstand the load again. Fig. 7 shows the process of testing the specimen.

While Fig. 8.a and Fig. 8.b show an example of the failure of the test specimen during an ultimate load is reached.

Figure 7. Testing process of the Meranti Timber Beam-Column Joint Connection.

(a). Meranti timber specimen.

(b). Mersawa timber specimen

Figure 8. Failure mode of the specimens.

Test results for the Beam-Column Timber Joint Connections (six specimens) are shown in Fig. 9 (Meranti Timber specimens), Fig. 10 and Fig. 11 (Mersawa Timber specimens). The test results are a curve of the load vs deformation, which represents the behavior and capacity of the beam-column joint.

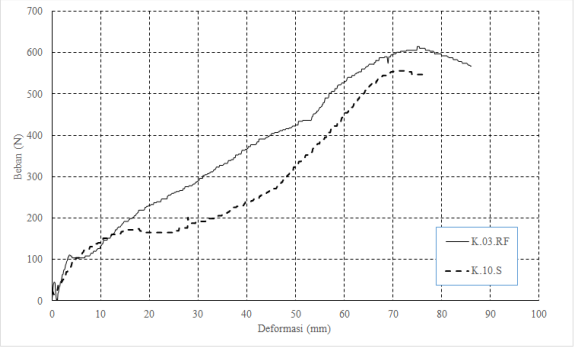

Figure 9. Meranti wood connections test results: Comparison of Load-Deformation Relations curve for the standard type and the PRP type connections.

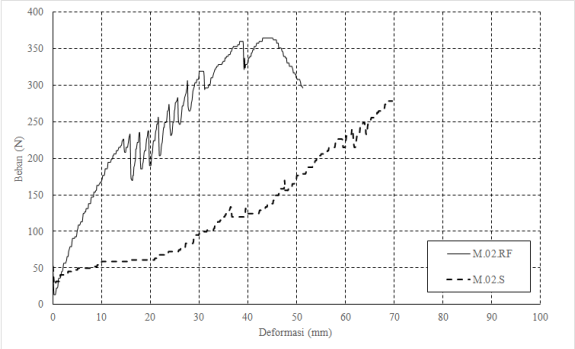

Figure 10. Mersawa wood connections test results: Comparison of Load-Deformation Relations curve for the standard type and the PRP type connections, Specimen M.02.

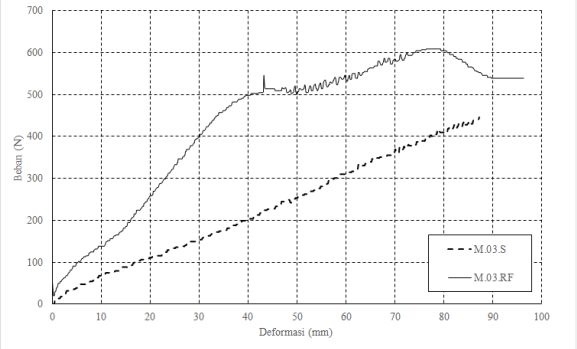

Figure 11. Mersawa wood connections test results: Comparison of Load-Deformation Relations curve for the standard type and the PRP type connections, Specimen M.03.

Fig. 9, Fig. 10, and Fig. 11 generally show test results of the load vs. deformation curve for both Meranti (Fig. 9) and Mersawa specimens (Fig. 10 and Fig. 11), which indicates that the overall capacity of the standard type lower than the PRP type.

Table 1 shows results of the timber joints made with Meranti wood, which are the idealization of the load vs. deformation curve, while Table 2 shows results of the timber joints made with Mersawa wood, i.e with reviewing the conditions of the proportional limit and ultimate limit. Method that used to determine both the proportional (P_y) and ultimate (P_u) limit loads using Yasumura and Kawai (Y&K) Method, namely a method for determining proportional limit loads and ultimate limit loads, specifically for wood material (Munoz *et al*. 2010).

Proportional limit load is a condition when there is a change from elastic to plastic behavior, while ultimate limit load is a peak load or peak capacity of the joints. Displacement ductility ratio is calculated using Equation 1.

Table 1. Summary of experimental results: Meranti specimens.

Specimen	P_y	D_y	P_u	D_u	μ
	(N)	(mm)	(N)	(mm)	(mm/ mm)
S (Standard)	526.49	66.40	555.86	72.60	1.09
RF (PRP)	571.68	66.10	605.57	74.40	1.13
%-difference	8.58%		8.94%		2.94%

Note: S = standard type, RF = PRP type.

Table 2. Summary of experimental results: Mersawa specimens.

Specimen	P_y	D_y	P_u	D_u	μ
	(N)	(mm)	(N)	(mm)	(mm/ mm)
S (Standard)					
M.02.S	178.78	51.10	278.35	68.90	1.35
M.03.S	350.76	67.50	441.28	86.70	1.28
Average	264.77		359.82		1.32
RF (PRP)					
M.02.RF	224.04	15.50	364.35	43.10	2.78
M.03.RF	468.44	35.90	604.22	79.40	2.21
Average	346.24		484.29		2.50
%-difference	30.77		34.59		89.62

Note: S = standard type, RF = PRP type.

The test results show that the beam-colum joint connection with the strengthening of the PRP type is more ductile than the Standard (S) type connection, both for Meranti and Mersawa wood connections.

In general the beam-column Mersawa timber joint connection type of PRP produce higher strength capacities ranging from 30.77% to 34.59% compared to the standard beam-column joint connection (in terms of Proportional Limit and Ultimate Limit Loads), while the beam-column Meranti

timber joint connection of type PRP also produce higher than standard type ranging from 8.58% to 8.94%.

The stiffness capacity, in term of Displacement Ductility Ratio of the Mersawa PRP type is 89.62% higher than standard type, while the Meranti PRP type is 2.94% higher than standard type.

Conclusions

This result indicates that the use of nails and modified-washer make a positive contribution to improving the performance of the beam-column joint connections, in terms of strength capacity (both of proportional limit and ultimate limit loads) and stiffness capacity (displacement ductility ratio). Meranti beam-column timber joint is more brittle than Mersawa beam column timber joint, it has an impact on the results. PRP-type of Mersawa timber connection produces a higher ductility than the standard type. While the PRP-type of Meranti timber connection produces a similar ductility to the standard type.

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Furfuryl Alcohol Treatment of Bamboo Betung (*Dendrocalamus asper* Backer ex K. Heyne) Strips

Krisdianto, Peter Vinden, and Simon Przewloka

Abstract

Based upon successful chemical modification of wood, bamboo strips were subjected to furfurylation treatment. Furfurylation in the mean of impregnating furfuryl alcohol to the bamboo and then heated at 100°C for 24 hours to produce solid polymeric resin. The success of furfurylation is assessed by uptake and the weight percentage gain of furfurylated bamboo strips. However, the treatability of dry bamboo strip is relatively poor. This paper studies the furfurylation process of bamboo betung (*Dendrocalamus asper* Backer ex K.Heyne) strips by soaking, vacuuming and the combinations thereof. Results showed that soaking bamboo strips for two days after vacuum treatment achieved optimum uptake of furfuryl alcohol solutions and gave rise to an 80% weight gain. Catalyst addition during furfurylation did not increase the weight percent gain. Water was an active solvent carrier for achieving higher weight gains.

Keywords: Bamboo strips, furfurylation, uptake, percentage weight gain, catalyst.

Introduction

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

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

Despite its many advantageous properties, bamboo is liable to attack by brown rot, white rot and soft rot fungi and by insects such as beetles and termites (Liese 1959; Kumar *et al.* 1994). Non-treated bamboo has an average life of fewer than 1~3 years when exposed to the atmosphere and soil. When used indoors, bamboo may be expected to last 4~7 years (Liese 1980), and therefore treatment is regarded as a necessity (Farelly 1984).

Environmental concerns regarding the use of chemicals for wood preservation has generated interest in alternatives such as chemical modification (Hill 2006). Wood modification is a means of altering the material to overcome one or more of its disadvantages. One of the chemical modification processes for wood is furfurylation (Jones 2007). Furfurylation is based upon the reaction of wood with the bio-chemical furfuryl alcohol at the cell wall level. The processes have significantly improved wood properties, protecting against decay, decreased hygroscopicity and improved dimensional stability (Jones and Hill 2007). Modification of wood with furfuryl alcohol resin is known to be an efficient method of obtaining wood products with high dimensional stability, high durability and resistance to alkali and acids (Homan and Jorissen 2004).

Furfuryl alcohol was first used to treat wood by Goldstein (1955), who impregnated wood with a mixture of furfuryl alcohol, water and a suitable acid catalyst. The impregnated wood was then heated at 100ºC for 24 hours to produce a solid polymeric resin. Goldstein (1955) demonstrated that zinc chloride and organic acids such as citric acid are suitable catalysts for furfurylation. Prior to curing, wood specimens were wrapped in aluminium foil and allowed to equilibrate for a day to enable migration of the solution through the structure by capillary action and diffusion (Goldstein 1955).

Furan derivatives are necessary industrial and manufacturing chemicals. They are used as solvents, reagents for chemical synthesis and as monomers in polymerization (Kim *et al.* 1998). The two intermediates employed for the commercial manufacture of furan resin are furfural and furfuryl alcohol. Furfural is produced from renewable agricultural by-products, and furfuryl alcohol is manufactured from furfural by a simple hydrogenation

process (Schultz 1990). Furfuryl alcohol is the most important derivative of furan and is primarily used in the synthesis of adhesive polymers (McKillip and Sherman 1980). Furfuryl alcohol is a monomer of oligomeric furfuryl alcohol resins and is widely used as a highly durable thermosetting metal coating (Kim *et al.* 1998).

Furfuryl alcohol has a boiling point of 170°C and a molecular weight of 98.10. It is a colourless to golden clear liquid with a pungent smell that darkens rapidly in the presence of air due to oxidation. Furfuryl alcohol is produced by a partial reduction of furfural, which is derived from pentose (C_5 sugars) containing plant material. Currently, furfuryl alcohol is produced from sugar cane bagasse, corn cobs, oat hulls, rice hulls, straw and wood waste.

Furfuryl alcohol has numerous industrial applications and is used extensively as a selective solvent for lubrication oil refining and extraction of unsaturated C_4 and C_5 hydrocarbons. It is widely used in foundries and the manufacture of anticorrosive paints. Furfuryl alcohol has been used for the preparation of materials requiring stability, acid, alkali and solvent resistance at relatively low costs. Some of the elements that have been manufactured using furfuryl alcohol resin include tabletops, ceramic sinks, furfuryl alcohol resin cement for bonding corrosion-resistant tiles or bricks, tank coatings, brick linings, bonding of glass fibre mats, adhesives in aircraft industries, manufacture of storage battery cases, solvent for cellulose esters, vinyl compounds, many natural gums and phenolic resins (Schmitt 1974).

Furfurylation with or without catalyst has been reported to enhance several properties of wood, such as protection against decay (as a result of reduced moisture and altered wood chemistry), increased dimensional stability (as a result of polymerization in the cell lumens), golden brown appearance and decreased hygroscopicity (Jones and Hill 2007). For wood, various treatment methods have been studied for furfuryl alcohol impregnation, including the full cell (Bethell) and empty cell (Lowry) processes (Goldstein 1955). Several different catalysts have been tested for the use with furfuryl alcohol. Goldstein and Dreher (1960) examined numerous catalysts and found a small number to have the potential use (zinc chloride, maleic acid, malonic acid, tartaric acid, malic acid and citric acid). Catalyst concentration of 5% was determined as sufficient for wood furfurylation.

Wood treated with furfuryl alcohol requires curing. This involves placing the samples in a uniformly heated environment for a set period. Curing time for furfurylation can be defined by three steps: polymerization of the alcohol resin, early resin curing stage (rubber) and slow curing stage (brittle). The initial moisture content of wood also affects the furfurylation process in wood. Schultz (1990) suggested that the water content of the cell walls has an effect upon the process.

Furfurylation has not been investigated as a possible treatment of bamboo. Recommended methods for liquid penetration of bamboo include soaking and vacuum with less pressure (Kumar *et al.* 1994). Vascular bundles which contain meta-xylem vessels and thickly walled fibre play an essential role in liquid penetration in bamboo. Unlike wood, there are no ray cells in bamboo, and the horizontal movement of liquid from vessels into neighbouring parenchyma tissue and fibres is by diffusion (Liese 1980). The diffusivity of furfuryl alcohol may facilitate the movement of the chemical from the lumens in vessels into surrounding tissues. This paper examines the possible applicability of furfurylation to bamboo strips.

Materials and Methods

Furfuryl Alcohol Treatment of Bamboo Strips

Strips of bamboo betung (*Dendrocalamus asper* Backer ex K. Heyne) were prepared from the outer, middle and inner regions of culm wall. Strips with the dimension of 5 x 10 x 75 mm were prepared free from inner and outer layers. Ninety strips were grouped into three treatments (soaking, vacuum and vacuum followed by soaking) and three regions (outer, middle and inner), each with ten replicates. To avoid bias, matched samples were prepared from one internode of ten bamboo culms.

After weighing, the strips were treated using 95% furfuryl alcohol by soaking, vacuum and a combination of vacuum and soaking. The soaking time was 1, 2, 3, 4 and 5 days. The vacuum method was applied by loading the strips in a vacuum glass container and evacuated in -85 kPa (gauge) for 10, 15 and 30 minutes before flooding with the 95% furfuryl alcohol. For the combination method, strips were evacuated for 10 minutes followed by soaking in solution for 1, 2, 3, 4 and 5 days.

The Effect of Catalyst and Carrier upon Furfurylation

Bamboo betung (*Dendrocalamus asper* Backer ex K. Heyne) blocks (10 x 10 x 10 mm) were prepared from one internode of bamboo culm (Fig. 1) to avoid bias. Two tropical hardwood species (*Anthocephalus chinensis* and *Calophyllum* sp.) with a slightly lower and higher density than bamboo were subjected to an identical process for comparison. All samples were oven-dried to allow calculation of the initial weight of non-modified blocks prior to treatment with furfuryl alcohol.

All blocks were treated using vacuum (-85 kPa) for 10 minutes, followed by two days soaking in a mixture of furfuryl alcohol and catalyst. A mixture of 90% furfuryl alcohol, 5% water, and 5% catalyst was combined on a weight/weight basis for the 5% catalyst loading solution. Catalysts used in this study included citric acid, zinc chloride, and maleic acid.

To determine the effectiveness of the ratio of water: furfuryl alcohol for bamboo furfurylation, four solutions of water and furfuryl alcohol (90:10; 50:50; 30:70; 15:85) were examined. Eight replicates were employed for each treatment. After treatment, all samples were wrapped in

aluminium foil and cured in an oven at 103ºC for 16 hours. The aluminium foil was then removed, and the specimens were post-cured for further eight hours at 103ºC. All surfaces of the blocks were sanded slightly in order to avoid the effect of poly-furfuryl alcohol coating (Westin *et al.* 2004). A critical parameter of furfurylation is the polymer mass after curing (Venas and Rinnan 2008). The polymer mass was evaluated by the weight percent gain.

Results and Discussion

Furfuryl Alcohol Treatment of Bamboo Strips

The average furfuryl alcohol uptake with soaking time is shown in Table 1. Generally, the average liquid uptake increased with increasing soaking time. Strips taken from the middle region achieved an average uptake of 24.1 kg/m^3 after one day, followed by 28.9 kg/m^3 after a further day. The highest uptake reached 34.3 kg/m^3 was achieved after five days of soaking.

Bamboo strips taken from the outer regions of the culm wall achieved higher uptake than the middle and inner strips. Strips taken from the outer region achieved an average uptake of 25.9 kg/m^3 after one day, followed by 31 kg/m^3 after two days and reached an uptake of 40.8 kg/m^3 after five days soaking. Two-way analysis of variance showed soaking time (1~5 days soaking) was significantly different ($F = 161.5$, $p = <0.001$) and similarly, strip portion (outer, middle and inner) was significantly different ($F = 190.4$, $p = <0.001$). The furfuryl alcohol uptake was rapid on the first day, followed by a slight uptake increase with subsequent soaking. The rapid initial uptake presumably occurred by capillary flow through vessels.

Furfuryl alcohol uptake after soaking varied between strips (outer, middle and inner). The outer strips demonstrated the highest uptake, while the inner strips had the lowest uptake. It is postulated that liquid penetrates mainly through vessels which more frequently exist in the outer region than the middle and inner sections (Liese 1980). The average furfuryl alcohol uptake after vacuum treatment is shown in Table 2.

Table 1. Furfuryl alcohol uptake of bamboo strips after soaking.

Bamboo Strips	Soaking time									
	1 day		2 days		3 days		4 days		5 days	
	x	*sd*	*x*	*sd*	*x*	*sd*	*x*	*sd*	*x*	*sd*
Outer	25.99	1.44	31.01	1.26	35.41	1.52	39.16	2.24	40.81	2.58
Middle	24.14	1.30	28.85	1.78	31.84	2.34	33.39	1.83	34.32	2.21
Inner	20.31	1.85	23.08	0.85	26.35	1.51	28.47	1.36	29.87	1.88

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

Table 2. Furfuryl alcohol uptake in bamboo strips after vacuum treatment.

Bamboo Strips	Vacuum time							
	10 minutes		15 minutes		20 minutes		30 minutes	
	x	*sd*	*x*	*sd*	*x*	*sd*	*x*	*sd*
Outer	113.42	2.69	126.45	4.23	121.51	2.42	122.72	2.78
Middle	107.30	3.12	110.14	4.42	119.72	2.10	116.27	2.33
Inner	92.49	1.07	89.53	1.84	90.67	1.25	93.59	1.37

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

Table 3. Furfuryl alcohol uptake in bamboo strips after an initial vacuum followed by soaking.

Bamboo Strips	Soaking time											
	0 day		1 day		2 days		3 days		4 days		5 days	
	x	*sd*	*x*	*sd*	*x*	*sd*	*x*	*sd*	*x*	*sd*	*x*	*sd*
Outer	118.1	5.9	121.9	3.2	131.9	2.2	134.2	2.4	134.9	2.9	135.9	2.8
Middle	106.1	3.6	118.3	4.7	134.9	1.1	137.3	2.2	140.5	1.4	141.9	1.4
Inner	93.5	1.8	104.1	4.5	116.4	3.6	117.9	3.2	119.8	3.1	120.6	3.1

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

Liquid uptake of bamboo strips after vacuum treatment was substantially higher than that achieved by soaking. The average uptake of strips vacuum treated was about three times higher than those soaked. The average uptake of the middle strips was 107.3 kg/m^3 with vacuum treatment compared to 34.3 kg/m^3 after five days soaking. In the culm, there are air bubbles that restrict the entry of solution. A vacuum condition was able to remove these air bubbles and allow higher uptake partially. The liquid uptake in bamboo strips slightly increased with increased vacuuming time. Two-way analysis of variance showed vacuuming time (10~30 minutes) was significantly different ($F = 37.6$, $p = <0.001$) and similarly, strip portion (outer, middle and inner) was also significantly different ($F = 1305.6$, $p = <0.001$). Strips from the inner region of the culm gain less liquid than the outer and middle regions. This is attributed to the smaller number of vessels found in the inner section.

The average furfuryl alcohol uptake in bamboo strips after an initial vacuum followed by soaking is shown in Table 3. Liquid uptake of bamboo strips after an initial vacuum followed by soaking treatment was considerably higher than that achieved by soaking and vacuuming alone. The average uptake of middle strips was 141.9 kg/m^3 after 10 minutes vacuuming followed by five days soaking, compared to only 34.3 kg/m^3 uptake after five days soaking only. The uptake improvement after vacuuming and soaking treatment is presumably caused by liquid penetrating through vessels during vacuuming followed by solution moving into the surrounding vessels by diffusion.

The results revealed that two days soaking after vacuum treatment achieved an optimum level of uptake in the bamboo strips. This confirms the findings of Skewes (2004), who reported that wood containing furfuryl alcohol continues to swell for up to 48 hours. Furfuryl alcohol expanded in the bamboo cell wall, allowing more liquid penetration.

Two-way analysis of variance showed soaking time after vacuum treatment (0~5 days) was significantly different ($F = 290.58$, $p = <0.001$) and similarly, strip portion (outer, middle and inner) was also significantly different ($F = 432.4$, $p = <0.001$).

The Effect of Catalyst and Carrier Upon Furfurylation

The average weight percent gain of sample blocks after furfurylation is shown in Fig. 1. The graph in Fig. 1 shows bamboo block samples with catalyst addition gained less weight than without catalyst. Two tropical hardwoods increased various weight percentage with catalyst addition. *Anthocephalus* woodblocks, which were less dense than bamboo, gained up to 142% weight after furfurylation with maleic acid as a catalyst. The high percentage gain (more than 100%) is due to polymerization of furfuryl alcohol not only in the wood cell wall but also in the void volume between cells. *Calophyllum* woodblocks which were denser than bamboo gained 87.4% weight with maleic acid addition. In hardwood block samples, the use of maleic acid as a catalyst achieved the highest percentage gain.

Bamboo blocks gained 2~3% weight after furfurylation with the catalyst. Visual investigation showed dark solution lying upon the surface of the blocks (Fig. 2A). The blocks cross-sections were filled with a coloured solution, but the surrounding ground parenchyma tissue appeared clear from the solution (Fig. 2B). Furfuryl alcohol solutions with catalyst addition did not penetrate the bamboo blocks, due to premature polymerization of furfuryl alcohol blocking further penetration of the solution.

Visual examination of furfurylated blocks shows dark cured resin appearing not only in the vessels but also in the ground parenchyma tissue (Fig. 2). Water solvent carrier improved the diffusion rate, and as a result, the furfuryl alcohol penetrated more area of the bamboo blocks.

The average weight percent gain of sample blocks after furfurylation with different concentrations is shown in graph Fig. 3. The furfuryl alcohol mixed with water resulted in considerably increased weight percent gain in bamboo blocks. Bamboo blocks treated with 90% furfuryl alcohol gained the highest weight percentage (up to 84%). Weight gain decreased with lower furfuryl alcohol concentration. This trend was not noted in the hardwood blocks. In hardwood blocks, furfurylation without catalyst resulted in lower weight percentage gain. The higher weight gained by bamboo blocks is possibly due to more significant starch content in bamboo. The starch is decomposed by the heat during curing, creating an acidic environment which accelerates furfurylation.

Visual examination of furfurylated blocks shows dark cured resin appearing not only in the vessels but also in the ground parenchyma tissue (Fig. 4). Water solvent carrier improved the diffusion rate, and as a result, the furfuryl alcohol penetrated more area of the bamboo blocks.

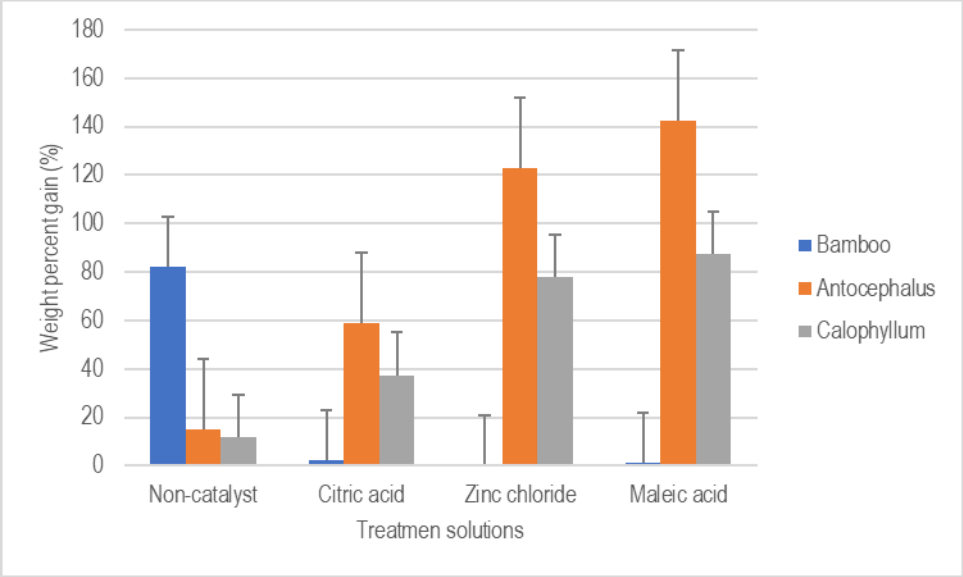


Figure 1. Weight percent gain in bamboo, *Anthocephalus* and *Calophyllum* blocks after furfurylation with catalyst.

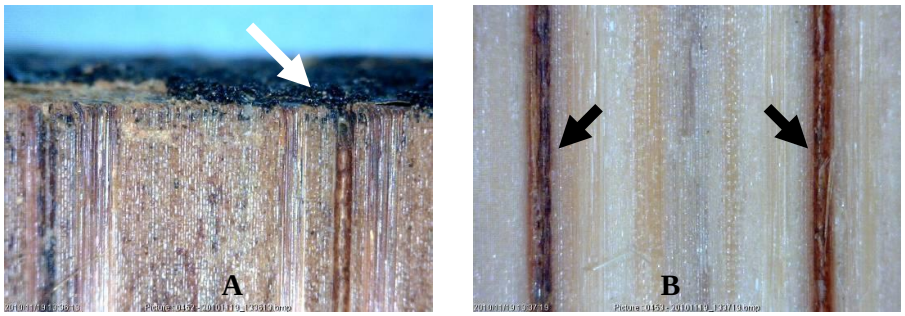


Figure 2. Visual examination of cured bamboo blocks.

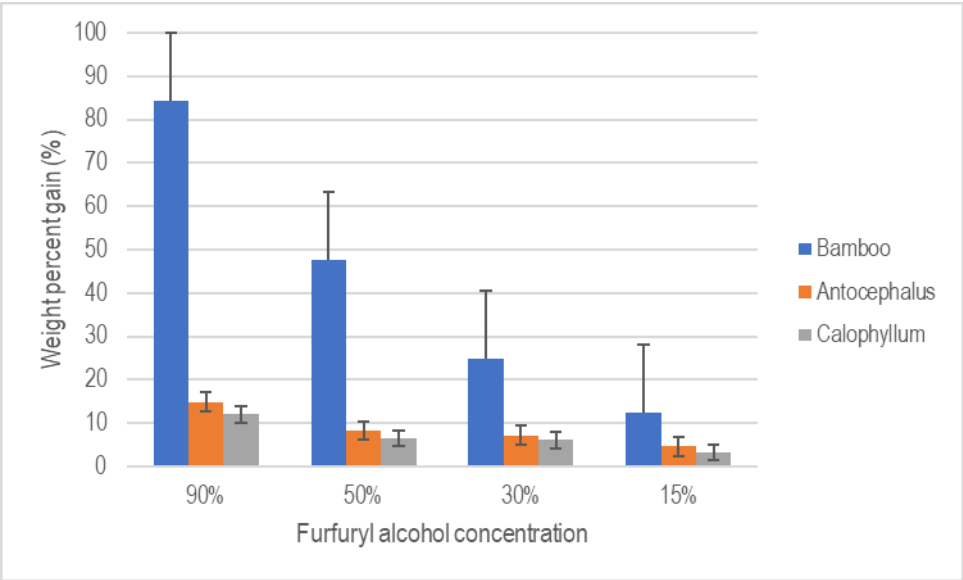
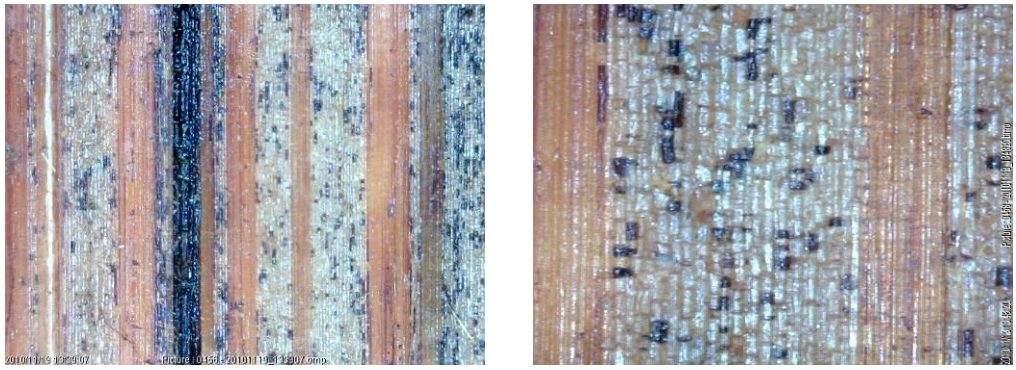


Figure 3. Weight percent gain in blocks with various furfuryl alcohol concentrations.



A **B**

Figure 4. Visual examination of furfurylated bamboo using water as a carrier solvent.

Conclusions

This study shows that the longer the soaking and vacuuming time, the higher the furfuryl alcohol uptake. The differences in uptake between strip portion (outer, middle and inner) show a clear trend that the outer strips have the highest uptake and the inner strips have the lowest uptake. They were soaking bamboo strips for two days after vacuum treatment achieved optimum uptake of furfuryl alcohol. The uptake improvement after vacuuming and soaking treatments is caused by liquid penetration through vessels during vacuuming followed by solution moving into the surrounding vessels by diffusion.

The addition of a catalyst to furfural alcohol significantly reduced the weight percent gain in bamboo, due to premature polymerization of the alcohol blocking further penetration. Addition of low concentrations (10%) of water to the furfuryl alcohol was optimum in achieving higher chemical uptake in bamboo.

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Effect of Distillation Tank Density and Storage Time on the Quality and Chemical Composition of Cajuput Oil

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Abstract

Cajuput oil is a commodity of non-timber forest product which is needed and potential to be developed in Indonesia. Therefore, further research on the factors of production and post-production are needed to produce optimum quality. In this study, the leaves of cajuput (*Melaleuca cajuputi* Powell) were distillated by water-steam distillation. This study evaluated effects of distillation tank density (60%, 70%, and 80%) and oil storage time (0 month, 1 month, 2 months, and 3 months) on physicochemical properties and chemical compositions of cajuput oils. The results showed that cajuput oils had a specific gravity of 0.915~0.923; optical rotation of (-2,10°) ~ (-1,20°); refractive index of 1.463~1.464; solubility in alcohol 1 : 1; cineole contents of 61.18~76.24%; clear to yellowish clear color; distinctive smell of cajuput and in accordance with SNI 06-3954-2006. The GC-MS analysis identified 24 of chemical components on the cajuput oils with main components were 1,8-cineole, α-pinene, and β-caryophylene. Tank density 70% to 80% with the oil storage time up to 3 months still gives the optimum quality and chemical compositions.

Keywords : cajuput oil, distillation tank density, storage time, physico-chemical properties, chemical composition.

Introduction

Cajuput oil is one of potential essential oil in Indonesia. Several plants species which producing cajuput oil and planted in Indonesia are *Melaleuca cajuputi* Powell and M. *leucadendron* Linn. (Widiyanto and Siarudin 2013). Other species which is being develop are M. *minor* Smith. and M. *viridiflora* Gartn. (Guenther 1987).

Cajuput oil production from natural forest in Maluku Archipelago with area of approximately 120,000 ha reached 196 ton/year and after economy crisis in Indonesia, it decreased to 100 ton/year. In Java, the states (Perum Perhutani) manage cajuput plants in the area of 18,000 ha with oil productivity of 300 ton/year, meanwhile Forestry Service of Yogyakarta Province manage cajuput plants in the area of 4,000 ha with oil productivity of 50 ton/year (Kartikawati and Rimbawanto 2014).

In fact, state production is insufficient to meet domestic demand. Cajuput oil packing industries or pharmacy industries information according to Kartikawati and Rimbawanto (2014) reported that state cajuput oil need reach up to 1,500 ton/year while the yearly supply is only 400 ton. The shortage of more than 1,000 ton is closed by importing eucalypt oil from China. Those problems could be overcome if the plants parts and processing factory is revitalized (Kartikawati and Rimbawanto 2014). There are some factors in oil processing which ascertain the quantity (yield) and quality of cajuput oil products. Factors influencing yield i.e. climate and soil, harvesting month, plants age, plants species, plants spacing, leaves condition, and oil processing. Factors influencing oil qualities i.e. plants species, leaves storaging method, tank filling which is related to leaves density in the tank, oil taking steps, and oil storage after processing (Kasmudjo 2011).

Related to the tank filling method especially cajuput leaves density in the distillation tank is if the leaves density in the tank is too high, it would prevent the steamed water to evaporate in the distillation process and makes the leaves wet which escalating the hydrolysis process and decreasing the oil quality. Moreover, the blocked steamed water would push to make way out and resulting in making uneven flow in the entire leaves. Some oils would stay in the leaves. Thus, the oil quality would decrease (Sumadiwangsa and Silitonga 1977). Leaves filling about half and a quarter of tank volume and adding the branch up to 20% would maintain the production of oil with high quality (cineole content up to 50%). In the factor of cajuput oil storage after processing, it could evaporate in room temperature without decomposing, being in the group of volatile oil (Kasmudjo 2011). Cajuput oil storage would evaporate the main components inside the oil (Guenther 1987). Turek and Stintzing (2012) analyze that essential oil composition is easily changed in the isolated oil storage. Volatile oil is susceptible to conversion and degradation reaction of oxidation process and polymerization caused by temperature, lights, and oxygen availability. Main components of cajuput oil are 1,8-cineolee, sesquiterpene alcohol globulol, viridiflorol, and spathulenol, and also minor components such as: limonene, β-caryophyllene, humulene, viridiflorene, α-terpineol, α dan β-selinene, and caryophyllene oxide (Brophy and Doran 1996). Quality decreasing caused by chemical changing would influence the pharmacological function of the cajuput oil (Turek and Stintzing 2012). Volatile oil which contains high monoterpene or oxide (cineole) has the storage time of 1~3 years (Anonymous 2014).

Due to those concerns, this study evaluated variations of distillation tank density 60%, 70%, and 80% of tank

volume, in the oil storage time after processing of 0 month, 1 month, 2 months, and 3 months to obtained optimal yield of cajuput oil quality and compositions.

Materials and Methods

Preparation and Essential Oil Samples

Fresh leaves of cajuput (*M. cajuputi* Powell) were obtained from RPH Menggoran Playen, Gunung Kidul, Yogyakarta, Indonesia. Leaves and terminal twigs were taken about 1 kg, and then the leaves are separated to the branches. Branches used should have the length of about 25 cm and diameter of 0.4 mm. Average proportion of leaves and branches obtained from this method was 78% : 22%. In order to get the optimum proportion of leaves and branches (80% : 20%), leaves mass was added by 2% and branches mass was reduced by 2%. Distillation tank capacity used in this research was 10 kg. Thus, to make 60% of tank density, it was filled up to 6 kg, to make 70% of tank density, it was filled up to 7 kg, and to make 60% of tank density, it was filled up to 8 kg. Water-steam distillation method was used in this study. Cajuput oils were produced put in dark glass bottle and storage for 3 months at close storage with room temperature (±28°C) and humidity of 84%.

Physicochemical Properties

Physico-chemcial properties testing of cajuput oils were done based on SNI 06-3954-2006. Analyses determined color, odor, specific gravity at 20°C, refractive index at 20°C, solubility index in 70% alcohol, and optical rotation. Oil color was analyzed based on visual observation, and odor was evaluated by the direct smell of paper strip containing the oil.

Optical rotation of oil was measured by disk polarimeter (WGX-4, Shanghai Benson Instrument Co. Ltd, Shanghai, China). Optical rotation is expressed in degrees of circumference (º). Optical rotation *dextro* is a positive sign (+) and optical rotation *levo* is a negative sign (-).

Specific gravity was measured by pycnometer based on the weight ratio of oil and water in the same volume at the same temperature. This test used 5 ml volume of pycnometer. The empty pycnometer weighed (m), then it was filled with distilled water (avoiding any air bubbles) and weighed (m_1). Then pycnometer was washed with ethanol and subsequently diethyl ether, and dried. This pycnometer filled with oil and weighed (m_2). The specific gravity of oil was obtained using the following equation:

$$d^{20} = d^t + 0.0007 \times (t-20)$$

where d^{20} is specific gravity at 20°C, t is ambient temperature (ºC), 0.0007 is correction factor.

$$d^t = (m_2 - m)/(m_1 - m)$$

where d^t is specific gravity at ambient temperature (t°C), m (g) is weight of empty pycnometer, m_1 (g) is weight of pycnometer contained water at t°C, m_2 (g) is weight of pycnometer contained oil at t°C.

Refractive index was determined by handy refractometer (N-3000e, Atago Co., Ltd, Tokyo, Japan). The refractive index was calculated by following equation:

$$n_D = n^t_D + 0.0004 (t-20)$$

where n_D is index value at 20°C, n^t_D is index value at ambient temperature (t°C), 0.0004 is correction factor.

Solubility in 70% alcohol was estimated based on the volume ratio of oil to 70% alcohol. One ml of oil is put in 10 ml volumetric glass and 1 ml of 70% alcohol was added, then solution becomes clear after mixing well. If solution is not clear, 1 ml of 70% alcohol is further added until clear solution is obtained. The results are expressed as follows:

Solubility in 70% alcohol = (1 ml of oil) : (ml of 70% alcohol added)

GC-MS Analysis

Cajuput oil chemical composition were analyzed by GCMS-QP2010S SHIMADZU with the specification as follows: AGILENT HP 5MS column (0.25 mm id x 30 m, film thickness of 0.25 nm), carrying gas is Helium, and ionization EI 70 eV, oven column temperature 70°C, injection temperature 310°C, injection mode is splitless, sampling time 0.20 minutes, electric current control mode was pressure of 13.7 kPa, total current is 60 mL / minute, current column: 0.50 mL / minute, with linear velocity of 25.9 cm / second. Oven temperature was programmed from 70°C (5 minutes on hold) to 300°C (19 minutes on hold), and from 100~230°C every 15°C/minute raised was hold on 5 minutes. Chemical compositions were identified base on WILEY 229 data base library and were compared with some literatures.

Statistical Analysis

All experiments were replicated three times, and the data were averaged. The results were tested by two-way ANOVA. Significant differences between means were determined by Honestly Significant Different (HSD) test. Value of $P < 0.05$ were considered statistically significant.

Results and Discussion

Physicochemical Properties

Physicochemical properties of cajuput oil were evaluated including color, smell, specific gravity, optical rotation, refractive index, alcohol solubility ratio, cineole content, and were presented in the Table 1. Physicochemical variance analysis of Cajuput oil in this study were presented in Table 2.

Table 1. Physicochemical properties of Cajuput Oils

Sample	Specific Gravity	Optical rotation(°)	Refractive Index	Alcohol solubility ratio	Cineole Content (%)	Color	Smell
K_1L_0	0.917	-2.10	1.463	1 : 1	67.68	Clear – Yellowish Clear	Original Cajuput Smell
K_1L_1	0.921	-1.70	1.464	1 : 1	67.08		
K_1L_2	0.921	-1.37	1.464	1 : 1	76.24		
K_1L_3	0.922	-1.23	1.464	1 : 1	62.34		
K_2L_0	0.915	-2.23	1.463	1 : 1	65.95		
K_2L_1	0.920	-1.83	1.463	1 : 1	66.37		
K_2L_2	0.920	-1.33	1.463	1 : 1	74.75		
K_2L_3	0.922	-1.40	1.464	1 : 1	61.18		
K_3L_0	0.920	-2.00	1.463	1 : 1	68.80		
K_3L_1	0.922	-1.37	1.464	1 : 1	68.86		
K_3L_2	0.923	-1.20	1.463	1 : 1	76.03		
K_3L_3	0.923	-0.90	1.464	1 : 1	64.24		
Total Mean	0.921	-1.56	1.464	1 : 1	68.29		

Notes:
K_1: Tank density 60%, K_2: Tank density 70%, K_3: Tank density 80%, L_0: Oil Storage Time 0 month, L_1: Oil Storage Time 1 month, L_2 : Oil Storage Time 2 months, L_3: Oil Storage Time 3 month.

Table 2. Variance analysis of Cajuput Oil

Variance	Specific density	Optical rotation (°)	Refractive index	Solubility in 70% alcohol
Tank density (K)	6.41 **	1.60 ns	0.68 ns	- ns
Oil Storage Time (L)	10.74 **	7.16 **	0.67 ns	- ns
Interaction (K*L)	0.71 ns	0.12 ns	0.09 ns	- ns

Notes:
ns: not significant *:significant ** : very significant

Color and Smell

Color test result according to tank density and storage time entirely showed the same clear-yellowish color and is defined as good. This condition is relevant to the standard of SNI that is clear to yellowish green. Essential oils in this study have smell same with original cajuput smell. This condition is relevant to the standard of SNI that is original cajuput smell (Anonymous 2006).

Specific Gravity

Specific gravity of the cajuput oil is varied by the different factors of tank density and oil storage time about 0.915~0.924 with the mean of 0.921 and it is defined as high and good. The average value of this specific gravity is relevant to the quality standard of Indonesia National Standard (SNI) which is about 0.900~0.930 (Anonymous 2006).

According to the factor of leaves density in the distillation tank of 60%, 70%, and 80%, the average value of specific gravity are 0.920; 0.919; and 0.922 respectively. The density of 80% results the highest specific gravity and followed by 60% and 70%. It is presumed that there is a difference of the weighed fraction of chemical component contents in the cajuput oil at the different factors of tank density. The more materials are processed, the more weighed fraction of chemical composition contents is contained in the material. Specific gravity is defined by the amount of oil components. The more long chain components such as sesquiterpen (C_{15}) and oxygen based components or oxygenated hydrocarbon is processed, the higher medium density or the specific gravity of the volatile oil processed (Ariyani *et al*. 2008). Oil of the distillation tank density of 80% contains the highest sesquiterpen and oxygenated hydrocarbon contents i.e. 92.83%, while on the density of 60% is 91.86%, and on the density of 70% is 90,90%. There are 16 chemical components in cajuput oil which are included in the group of sesquiterpen and oxygenated hydrocarbon, i.e.: butanoic acid, benzene, 1,8-cineole, 4-terpineol, α-terpineol, α-terpinyl acetat, viridiflorol, caryophyllene oxide, nerolidol, β-caryophyllene, α-humulene, β-gurjunene, β-selinene, α-selinene, α-caryophyllene, and β-elemene.

According to the factor of oil storage time of 0, 1, 2, and 3 months, the average value of specific gravity are 0.917; 0.921; 0.922; and 0.922, respectively. Specific gravity value is also increase gradually from the storage time of 0~3 months. It is presumed that the presence of oxidation and polymerization cause the average value of cajuput oil specific gravity raise from the storage time of 0~3 months. Oxidation of volatile oil could decrease the amount of chemical components in the oil (Ketaren 1985). Essential oil

composition is easily changing in the process and the isolated oil storage, where the external factors such as temperature, lights, and oxygen availability influence the changing of chemical composition process (Turek and Stintzing 2012). The amount of hydrocarbon component which has low saturation point is easily decreased in the room temperature storage (Najafian 2014). It is presumed that long components and components which has low saturation point is more resistant of those chemical reaction. The more long chained components such as *sesquiterpen* (C_{15}) and/or oxygen based components, the higher medium density or specific grafity of volatile oil would be (Ariyani *et al.* 2008). In this cajuput oil process, sesquiterpene and *oxygenated hydrocarbon* contents tends to increase on the storage time of 0 to 3 months by 90.03%, 91.53%, 93.77%, and 92.13% respectively. Terpene containing oil if is stored at the long period of time would form a certain resin or usually called as resinification or polymerization (Ketaren 1985). The resinous content presumes to take a certain part in the increasing of specific gravity. The higher specific gravity value due to the increasing of the resinous content would decrease the quality of the oil.

Variance analysis showed that the factors of tank density difference and oil storage time are significant, while the interaction is non-significant. The different variables of tank density and storage time factors influence the specific gravity, but if those factors are combined would not significantly influence the specific gravity. Storage time would be more important than tank density because ANOVAs value of the storage time is higher. Thus, it is important to pay attention to the oil storage time of cajuput oil processing to produce the optimal specific gravity.

Optical Rotation

Optical rotation of the cajuput oil is varied by the different factors of tank density and oil storage time about (-2.50°) ~ (-0.50°) with the average of -1.56° and is defines as high and good. The average value of optical rotation is relevant to the Indonesia National Standard i.e. (-4°) ~ (0°) (Anonymous 2006).

According to the factor of leaves density in the distillation tank of 60%, 70%, and 80%, the average value of optical rotation are -1.60°; -1.70°; dan -1.37°. The density of 80% results the highest specific gravity and followed by 60% and 70%. It is presumed that the value of optical rotation is linear to the value of specific gravity. The higher specific gravity, the higher optical rotation would be. The value of optical rotation is relevant to the value of the specific gravity according to the factor of tank density.

According to the factor of oil storage time of 0, 1, 2, and 3 months, the average value of optical rotation are -2.11°; -1.63°; -1.30°; and -1.18°, respectively or generally tends to increase. The value of optical rotation tends to increase by the increasing of specific gravity according to the factor of oil storage time of 0~3 months. The increasing of specific gravity is also increasing the value of optical rotation.

Variance analysis showed that the factor of oil storage time is very significant to optical rotation, while the factor of tank density and the interaction of both factors are non-significant. It is showed that the factor of oil storage time gives higher significant influence to optical rotation as one of cajuput oil quality value.

Refractive Index

Refractive index of the cajuput oil is varied by the different factors of tank density and oil storage time about 1.462~1.465 with the average of 1.464 and is defined as high and good. The average value of refractive index is relevant to the Indonesia National Standard i.e. 1.450~1.470 (Anonymous 2006).

According to the factor of leaves density in the distillation tank of 60%, 70%, and 80%, the average value of refractive index are nearly the same, i.e. 1.464; 1.463; dan 1.463 respectively. It is presumed that the value of refractive index is linear to the value of specific gravity and optical rotation. Oil which have high refractive index is usually also have high density (Effendi and Widjanarko 2014). The more long chain components such as sesquiterpen (C_{15}) and oxygen based components is processed, the higher medium density or the specific gravity of the volatile oil processed, and the incoming light would be more difficult to refract. This would cause the higher refraction index of the oil (Ariyani *et al.* 2008).

According to the factor of oil storage time of 0, 1, 2, and 3 months, the average value of refractive index are 1.463; 1.464; 1.464; dan 1.464, respectively and tends to increase slightly. It is presumed that resinification reaction cause the average value of specific gravity increase from the storage time of 0~3 months. The more long chain components such as sesquiterpen (C_{15}) and oxygen based components is processed, the higher medium density or the specific gravity of the volatile oil processed, and the incoming light would be more difficult to refract. This would cause the higher refraction index of the oil (Ariyani *et al.* 2008).

Variance analysis showed that the factor of tank density, oil storage time and both factors interaction is non-significant on the value of refractive index in the oil. It is showed that the factor of tank density and oil storage time give non-significant influence to the value of refractive index.

Solubility in 70% Alcohol

Alcohol solubility ratio of the cajuput oil by the different factors of tank density and oil storage time is entirely same for about 1 : 1 with clear condition which is defined as very good. The alcohol solubility ratio is relevant with the quality standard of SNI i.e. (1 : 1) ~ (1 : 10) (Anonymous 2006).

According to the factor of leaves density in the distillation tank of 60%, 70%, and 80%, the value of alcohol solubility content are entirely the same 1 : 1. Feasibility of

the oil to soluble in the alcohol is presumably caused by the high presence of oxygenated hydrocarbon components in the cajuput oil even though being processed at the different density, which is about 82.37~ 83.39%. It is relevant to the statement of Ketaren (1985), where oxygenated hydrocarbon components have higher solubility in liquid alcohol.

According to the factor of oil storage time of 0, 1, 2, and 3 months, the average value of alcohol solubility ratio are entirely the same for about 1 : 1. It is presumed that oil storage time factor is non-significantly influence the value of alcohol solubility ratio and the oil is evenly good. Oxygenated hydrocarbon components in cajuput oil is still high and not much change in the storage time of 0~3 months, and showed that the oil solubility in water is still good.

Cineole Content

Cineole content of the cajuput oil is varied by the different factors of tank density and oil storage time about 61~76% with the average of 68%. The average value of optical rotation is even higher than SNI i.e. 50~65% (Anonymous 2006).

According to the factor of leaves density in the distillation tank of 60%, 70%, and 80%, the average value of cineole content are 68.34%, 67.06%, and 69.48% respectively. According to Kasmudjo (2011) by filling half and a quarter (75%) volume of the distillation tank and adding the branches up to 20%, would produce the still high cineole content in the oil. It is presumed that by the tank density of 60%, 70%, and 80%, would show the still high cineole content at the main parameter of cajuput oil quality and relevant to the standard even if it is varied. Tank density of 80% produces the highest average of cineole content, while the lowest is 70%. It is presumed that it is caused by the higher oxidation occur at the tank density of 70% due to the leaves condition during distillation process or in the wetter storage condition. Oxidation of volatile oil could decrease the amount of chemical content in the oil (Ketaren 1985).

Table 3. T-test cineole content of Cajuput Oil related to tank density.

Variable	Mean	t test
K_1	0.68 ± 0.06	5.76 *
K_2	0.67 ± 0.06	
K_1	0.68 ± 0.06	-2.37 ns
K_3	0.70 ± 0.05	
K_2	0.67 ± 0.06	-6.08**
K_3	0.70 ± 0.05	

Notes:
K_1: Tank density 60%, K_2: Tank density 70%, K_3: Tank density 80

Table 4. T-test cineole content Cajuput Oil according to oil storage time.

Variable	Mean	t test
L_0	0.68 ± 0.01	0.134 ns
L_1	0.67 ± 0,01	
L_0	0.68 ± 0,01	-16.78 **
L_2	0.76 ± 0,01	
L_0	0.68 ± 0.01	20.98 **
L_3	0.63 ± 0.02	
L_1	0.67 ± 0.01	-14.28 **
L_2	0.76 ± 0.01	
L_1	0.67 ± 0.01	27.95 **
L_3	0.63 ± 0.02	
L_2	0.76 ± 0.01	19.97 **
L_3	0.63 ± 0.02	

Notes:
L_0: Oil Storage Time 0 month, L_1: Oil Storage Time 1 month, L_2: Oil Storage Time 2 months, L_3: Oil Storage Time 3 months.

T-test result at the factor of oil storage time to cineole content in Table 4 showed that storage time of 0 to 1 month is not significant, while storage time of 0 to 2, 0 to 3, 1 to 2, 1 to 3, and 2 to 3 is very significant.

According to the factor of oil storage time of 0, 1, 2, and 3 months, the average value of cineole content are 68%, 67%, 76% and 63% respectively. The highest cineole content is found at the storage time of 2 months which is 76%, while the lowest is at 3 months which is 63%. It is presumed that oxidation process in cajuput oil cause the cineole content in the oil become unstable in the storage time of 0 to 3 months. According to Ketaren (1985) oxidation process of volatile oil both cause smell changing and decreasing the amount of chemical components in the oil. Oxidation process in volatile oil mainly occur in the double chain reaction such as in 1,8-cineole ($C_{10}H_{18}O$), where 1,8-cineole is included in the group of *oxygenated hydrocarbon* component which contained terpene unit. In this research, the oil is kept in the minimum light room, but at the condition of room temperature and the storage cup still have room for air, oxygen (O_2) in the air would increase the occurrence of oxidation reaction and decreasing the oil quality. This occurrence is relevant to Najafian (2014) statement that the decreasing quality occurs higher in the volatile oil of *Melissa officinalis* during the storage at the room temperature to the temperature of 4°C and -20°C. Composition changing and the physical-chemical properties of volatile oil is generally more significant in the half-filled cup than only a little or none air room (Turek and Stintzing 2013).

T-test on the tank density factor to cineole content at the Table 3 showed that between the density of 70% and 80% is very significant, between the density of 60% and 70% in significant, while between the density of 60% and 80% is non-significant.

Table 5. Chemical composition of Cajuput Oil (%).

Compound	Chemical Formula	Retention Time	K_1				K_2				K_3				Mean
			L_0	L_1	L_2	L_3	L_0	L_1	L_2	L_3	L_0	L_1	L_2	L_3	
Butanoic acid	$C_6H_{12}O_2$	3.458	nd	0.41	nd	0.50	nd	0.66	0.17	0.91	nd	0.35	nd	0.39	0.28
α-thujene	$C_{10}H_{16}$	6.395	nd	nd	nd	Nd	nd	nd	0.18	nd	nd	nd	nd	nd	0.02
β-ocimene	$C_{10}H_{16}$	6.554	nd	nd	nd	3.01	3.93	nd	nd	3.07	2.75	2.87	nd	2.60	1.52
α-pinene	$C_{10}H_{16}$	7.002	4.02	3.15	1.87	Nd	nd	3.48	2.45	nd	nd	nd	2.11	nd	1.42
β-pinene	$C_{10}H_{16}$	8.337	3.05	2.56	1.66	2.66	3.11	2.80	2.05	2.73	2.38	2.80	1.87	2.27	2.50
β-myrcene	$C_{10}H_{16}$	8.705	2.91	2.09	1.24	2.04	2.68	2.33	1.54	2.16	2.05	2.21	1.47	2.09	2.07
p-cimene	$C_{10}H_{16}$	9.456	nd	nd	0.37	Nd	nd	nd	0.43	nd	nd	nd	nd	nd	0.07
Benzene	$C_{10}H_{18}O$	9.658	nd	nd	nd	Nd	nd	0.64	nd	nd	nd	nd	nd	nd	0.05
1.8-cineole	$C_{10}H_{18}O$	9.962	67.68	67.08	76.24	63.34	65.95	66.37	74.75	61.18	68.00	68.86	76.03	64.24	68.23
γ-terpinen	$C_{10}H_{16}$	10.587	0.86	0.45	0.3	0.34	1.33	0.69	0.57	0.61	0.86	nd	0.32	nd	0.53
α-terpinolene	$C_{10}H_{16}$	11.600	nd	nd	nd	Nd	nd	nd	0.25	nd	nd	nd	nd	nd	0.02
4-terpineol	$C_{10}H_{18}O$	14.740	0.86	0.77	0.57	0.84	0.85	0.85	0.62	0.90	nd	0.88	0.63	0.87	0.72
α-terpineol	$C_{10}H_{18}O$	15.037	10.94	10.47	7.95	11.97	9.91	9.17	6.91	10.9	10.48	10.69	8.35	12.17	9.99
α-terpinylacetat	$C_{12}H_{20}O_2$	19.859	1.87	2.69	1.89	3.42	3.23	4.49	3.3	6.23	1.80	2.91	2.17	3.76	3.15
β-caryophyllene	$C_{15}H_{24}$	21.989	6.15	6.12	3.30	9.65	7.13	6.36	3.26	7.03	8.45	6.65	3.39	8.12	6.30
α-humulene	$C_{15}H_{24}$	23.142	nd	nd	nd	Nd	nd	nd	nd	nd	nd	1.78	nd	nd	0.15
β-selinene	$C_{15}H_{24}$	22.918	nd	1.88	1.41	2.45	nd	1.77	1.35	2.24	nd	nd	1.44	2.37	1.24
α-caryophyllene	$C_{15}H_{24}$	23.322	1.68	nd	nd	Nd	1.89	nd	nd	nd	2.05	nd	nd	nd	0.47
β-elemene	$C_{15}H_{24}$	23.802	nd	1.73	1.38	Nd	nd	nd	0.86	1.36	nd	nd	0.99	0.50	0.57
β-gurjunene	$C_{15}H_{24}$	23.882	nd	nd	nd	Nd	nd	nd	nd	nd	1.18	nd	nd	nd	0.10
α-selinene	$C_{15}H_{24}$	24.027	nd	nd	1.25	Nd	nd	nd	0.91	nd	nd	nd	0.85	nd	0.25
Nerolidol	$C_{15}H_{26}O$	26.447	nd	nd	nd	0.78	nd	nd	nd	0.67	nd	nd	nd	nd	0.17
Viridiflorol	$C_{15}H_{26}O$	26.527	nd	0.61	0.56	Nd	nd	nd	0.41	nd	nd	nd	0.37	nd	0.16
Caryophyllene oxide	$C_{15}H_{24}O$	26.765	nd	nd	nd	Nd	nd	0.40	nd	nd	nd	nd	nd	nd	0.03

Notes:
nd: not detected, K_1: Tank density 60% , K_2: Tank density 70%, K_3: Tank density 80%, L_1 : Oil Storage Time 1 month, L_2 : Oil Storage Time 2 months, L_3 : Oil Storage Time 3 months

Table 6. Oxygenated hydrocarbon content in Cajuput Oil according to the factors of tank density and storage time (%).

Tank density	Storage time				Mean
	L_0	L_1	L_2	L_3	
K_1	81.35	82.03	87.21	79.84	82.61
K_2	79.94	82.58	86.16	80.79	82.37
K_3	80.28	83.69	87.55	82.03	83.39
Mean	80.52	82.77	86.97	80.89	82.79

Notes:
K_1: Tank density 60%, K_2: Tank density 70%, K_3: Tank density 80%, L_0: Oil Storage Time 0 month,
L_1: Oil Storage Time 1 month, L_2 : Oil Storage Time 2 months, L_3: Oil Storage Time 3 months.

Table 7. Hydrocarbon content in Cajuput Oil according to the factors of tank density and oil storage time (%).

Tank density	Storage time				Mean
	L_0	L_1	L_2	L_3	
K_1	18.66	17.98	12.78	20.15	17.39
K_2	20.05	17.43	13.85	19.20	17.63
K_3	19.72	16.31	12.44	17.95	16.61
Mean	19.48	17.24	13.02	19.10	17.21

Notes:
K_1: Tank density 60%, K_2: Tank density 70%, K_3: Tank density 80%, L_0: Oil Storage Time 0 month,
L_1: Oil Storage Time 1 month, L_2 : Oil Storage Time 2 months, L_3: Oil Storage Time 3 months.

Chemical Composition

GC-MS analysis identified 24 components in cajuput oil (Table 5). The most abundant component is 1,8-cineole with the average of 68.23%, while the lesser is α-thujene with the average of 0.02%. There are three main chemical components of cajuput oil i.e.: 1,8-cineole (68.23%), α-terpineol (9.99%), and β-caryophyllene (6.30%).

Chemical components of cajuput oil are divided into two groups, oxygenated hydrocarbon and hydrocarbon. There are 9 chemical components which are included into oxygenated hydrocarbon (C, H, dan O) group, i.e.: butanoic acid, benzene, 1,8-cineole, 4-terpineol, α-terpineol, α-terpinyl acetat, viridiflorol, caryophyllene oxide, and nerolidol (Table 6).

There are 15 chemical components which are included into hydrocarbon $(C_5H_8)_n$ groups, i.e.: α-thujene, β-ocimene, α-pinene, β-pinene, β-myrcene, p-cimene, γ-terpinen, α-terpinolene, β-caryophyllene, α-humulene, β-gurjunene, β-selinene, α-selinene, α-caryophyllene, dan β-elemene (Table 7). The relationship of hydrocarbon group and oxygenated hydrocarbon group is contradictory. Oxygenated hydrocarbon components influence the quality of cajuput oil include specific gravity, optical rotation, refractive index, and alcohol solubility ratio.

According to the factor of leaves density in the distillation tank of 60%, 70%, and 80%, the average value of oxygenated hydrocarbon are 82.61%; 82.37%; and 83.39% respectively. It is presumed that at the density of 80%, material condition is still relatively porous even though the material process is the most abundant. Thus, the distillation process would run optimally and the weighed fraction chemical components such as oxygenated hydrocarbon and sesquiterpene are abundant in the oil, while the lowest is at the density of 70%. It is presumed that that is caused by the higher oxidation process occur in the density of 70% due to the leaves condition in the process and the storage is wetter. Oxidation process in volatile oil could decrease the amount of chemical components of volatile oil (Ketaren 1985).

According to the factor of oil storage time of 0, 1, 2, and 3 months, the average value of hydrocarbon content are 19.48%; 17.24%; 13.02%; and 19.10% respectively. Hydrocarbon contents tend to decrease in the storage time of 0 to 3 months. It is presumed to be caused by the occurrence of oxidation process during the storage. Oxidation process in volatile oil could decrease the amount of chemical components of volatile oil (Ketaren 1985). This is relevant to the research by Rowshan *et al.* (2013) to the volatile oil of *Thymus daenensis* which showed that at the room temperature, hydrocarbon components proportion with low saturation point such as α-pinene, α-terpinene, myrcene, γ-terpinene, dan p-cymene decrease after storage time up to 3 months. The amount of hydrocarbon components with low saturation point would easily and highly decrease at the room temperature. This phenomenon is cause by evaporation, oxidation, and other unwanted changing in volatile oil during the storage (Najafian 2014). After 3 months storage time, oxygenated hydrocarbon components are started to decrease, and this presumed to be caused by oxidation. These components are more resistant to oxidation than hydrocarbon components which already decerase by the storage time of 0 month. This is relevant to the statement of Ketaren (1985) that oxygenated hydrocarbon components are more resistant and stable to oxidation and resinification, while the hydrocarbon components are the contrary.

Conclusions

Cajuput oils in this study have quality in accordance with SNI 06-3954-2006. These oils consist of 24 compounds with three major compounds of 1,8 - cineol , α - terpineol , and β - caryophyllene . Oxygenated hydrocarbon compound contents was between 82.37~83.39%, and hydrocarbons between 16.6~17.63%. Distillation tank density of 70% to 80% gives optimum yield and quality, whether storage time of cajuput oils until 3 months still have good chemical composition.

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Mycelia Growth of Shiitake (*Lentinula edodes)* on 4-Wood Species from Leguminaceae Family

Dahayu Ratnanindha, Johanes Pramana Gentur Sutapa, and Denny Irawati

Abstract

Cultivation of Shiitake mushroom (*Lentinula edodes*) in Indonesia has not been developed yet, due to its low productivity. It happens because of the limited information of the good ways on cultivation Shiitake in Indonesia. One of the factors that affect the growth of mushroom is the media. In Indonesia, the information about the media composition by using various species of wood have not been found yet. Therefore, this research was conducted to determine the effect of using different wood species to the growth of Shiitake. This study used four wood species from the leguminaceae family, those were: Gamal (*Glicidia sepium*); Johar (*Samanea saman*); Leucaena (*Leucaena leucocephala*); and Sengon (*Falcataria moluccana*). Those were used as the media by adding 12.5% (w/w) of rice bran and 6% (w/w) of $CaCO_3$, and adjusting the moisture content by adding the distillate water to 65, 70, and 75%. Then, the media was put in to the petridisk (ø 90 mm) and inoculated with Shiitake mushroom. During the mycelia growth, the length of mycelia was measured every 2 days until fifty days, and then glucosamine content was analyzed. In addition, chemical analysis was also conducted to each media. The results showed that different wood species resulted different chemical content of media, except the acid soluble lignin content. The growth rate of mycelia was affected by the moisture content of media, meanwhile the glucosamin content is influenced by the moisture content and various species of wood. The best combination to cultivate the Shiitake mushroom was by using media made of gamal with 70% moisture content. There were positive correlation among the ethanol-toluen extracts content and the mycelia growth, however there was negative correlation among the hemicelullose content the mycelia growth.

Keywords: Shiitake mushroom, Leguminoceae, mycelial growth, moisture content, media's chemical content.

Introduction

Shiitake (*Lentinula edodes*) is an edible mauhroom which is famous in Japan and may called also as Chinese Black Mushroom. This mushroom posses high trade value, however, unfortunately this Shiitake mushroom cultivation is not so well known in Indonesia and still needs many information on its development.

Generally mushroom's growth is affected very much by media condition such as, species which is used, moisture content, media composition, pH, and the environment condition of the media. The previous research results on ear-mushroom which was cultivated by 3 different species showed that very significant difference of micellium growth rate on each media from 3 different wood species was found (Irawati *et al*. 2012). For Shiitake it self, the optimum environment condition of growth site on temperature 5~35°C and 6~25°C for its fruiting bodies' growth, with optimum humidity among 95~100% (Widyastuti 2009). On the other hand, the proper media condition for Shiitake growth in Indonesia has not been known well. Recently, Shiitake is cultivated by people by using sengon media only, however, sometimes in the areas which are not the centre of sengon faces difficulties to get that wood. To reduce the dependency on sengon, therefore, other wood species which can be used as Shiitake media need to be found as an alternative.

In Indonesia some other wood species which are classified into family Leguminoceae are supposed to posses good characteristics for mushroom growth. Species from family Leguminoceae is usually easy to be found in community's yard especially in country side. Community plants species from Leguminoceae very much because they use the leaves as cattle feed or to fertilize soil. Information about wood species in Indonesia which is suitable for Shiitake mushroom cultivation and also physical condition of the media suh as moisture content have not been found yet. To support the development of Shiitake mushroom cultivation in Indonesia, therefore, research about the effect of various wood species especially which are classified as family Leguminoceae and optimum media's moisture content was conducted.

Materials and Methods

Fungus and Substrate Materials

Seed of Shiitake mushroom F1 (pure culture) was obtained from kelompok tani Sedyo Lestari, Bantul, Yogyaarta. Wood meal (9~80 mesh) of 4-wood species from family *Leguminaceae* which are Gamal (*Gliricidia sepium*), Johar (*Samanea saman*), Lamtoro (*Leucaena leucocephala*), and Sengon (*Falcataria moluccana*) were used as the basal cultivation substrates. Commercial rice bran (9~80 mesh size) was used as a nutritive additive. Chemical contents of wood and rice bran whic are used are shown on Table 1.

Table 1. Chemical componen content of 4-wood species and rice bran (%).

Chemical content	Sengon	Gamal	Johar	Lamtoro	Rice bran
Ethanol-toluene extractives	2.92±0.35	5.18±0.14	3.12±0.40	1.98±0.20	6.87±0.10
Holocellulose	80.78±0.63	83.85±0.12	84.97±0.55	83.56±1.07	81.58±0.82
α-cellulose	45.34±1.11	53.72±0.55	47.15±0.44	43.69±0.97	51.4±0.10
Hemicellulose	35.45±1.62	30.12±0.21	37.82±0.06	39.87±1.02	30.18±0.91
Klason lignin	23.30±0.30	27.47±0.39	20.16±0.79	22.00±0.28	36.73±0.20
Acid soluble lignin	2.83±0.08	1.35±0.05	2.60±0.01	1.48±0.03	1.8±0.034
Hot water soluble extractives	-	-	-	-	19.5±0.20

Wood meal and commercial rice bran were mixed in a weight ratio of 8:1. Moisture content (MC) of wood meal and rice bran was determined by 103±2°C and scaling them until we have constant weight. Calcium carbonate was added at a concentration of 6% (w/w) to adjust the pH of the cultivation substrate to between 6 and 7. MC of the substrate was adjusted to 65, 70, and 75% by adding tap water.

Chemical Components of Media

Chemical components of media was determined before cultivation. Extraction with organic solvents was performed to determine the quantities of holocellulose, α-cellulose, Klason lignin, and ash. Before chemical analysis, the samples were ground with a rotary speed mill and then sieved through 40~80 mesh size. Subsequently, the samples were dried in an oven at 45°C. Extraction with organic solvents was performed as follows: 5 g of sample was extracted with 120 ml of a mixture of ethanol and toluene (1:2, v:v) by a Soxhlet extractor for 6 h. The amounts of Klason lignin, holocellulose, and α-cellulose were determined by ordinary methods (Carrier *et al*. 2011; Irawati *et al*. 2013). To determine the ash content, a 1 g sample was heated in a muffle furnace at 600°C for 2 h, and then, after cooling in a desiccator, it was reheated at 600°C for 1 h. For all chemical component analyses, 3 replications were performed for each sample.

Mycelial Growth Rate

To measure the mycelial growth rate, 20 g of cultivation substrate was packed in a Petri dish (90 mm diameter) by following the method in published paper before (Irawati *et al*. 2012). Five replications were performed for each wood meal substrate. The substrates were subsequently autoclaved at 121°C for 20 min, and inoculated with mycelial plugs (5 mm diameter) of Shiitake, previously grown on potato dextrose agar medium. The culture was maintained at 25°C in the dark. Colony diameter was measured every 2 days in four directions until the mycelia reached the edge of each Petri dish.

Glucosamine Content

To determine the glucosamine content in the mycelium, 5 g of cultivation substrate was packed in a Petri dish (45 mm diameter). Chitin content in the mycelium was assayed by the method of Braid and Line (1981) and 3 repetitions were performed for each sample. To degrade fungal chitin into *N*-acetyl glucosamine, 1 g of the dry sample after 50 days of culture was hydrolyzed with 5 ml of 5 M HCl at 80°C for 20 h. The absorbance of the characteristic bluish green color was measured at 630 nm using a spectrophotometer. The calibration curve was drawn with N-Acetyl-D-(+)-glucosamine (Wako Pure Chemical Co., Japan) as a standard at concentrations of 0, 10, 20, and 30 mg/ml.

Results and Discussion

Chemical Contents of Media

Originaly, chemical content of media was different with chemical content of wood (Table 1), because there were rice bran and $CaCO_3$ addition into the media. Chemical content of media, then, was used as basic calculation and analysis. Table 2 shows that difference of wood species significantly affected to ethanol- toluene extractives content, hot water soluble extractive content, holocellulose, α-selulosa, hemicellulose, and Klason lignin, but it did not affect to acid soluble lignin. Media made of johar wood showed the highest ethanol-toluene extractives content (8.84%) and hot water soluble extractives content (16.3%) indicating that media made of johar wood is estimated to posses substrate contents such as waxes, fat, resin, tannin and eter components which are soluble into high polar and non-polar extractives and also high tannin content, gum, sugar, and colour substrates which are soluble in the water.

Holocellulose, α-cellulose, dan hemicellulose are carbohydrate polymer which are avaliable in the wood. The highest holocellulose and α-celulose which were found in media made of wood were 84.67% and 60.04%, respectively. Cellulose is the main polymeric component of the plant cell wall, the most abundant polysaccharide on earth, and an important renewable resource. Basidiomycetous fungi belong to its most potent degraders because many species grow in environment rich in cellulos (Baldrian and Valaskova 2008). Some edible mushroom species which are classified into Basidiumycetous. Hemicellulose content was calculated based on substraction between holocellulose content and α-cellulose. Table 2 shows that the highest hemicellulose content was found in media made of sengon wood 28.73%. During wood degradation, generally, hemicellulose which is

polysaccharide with short sugar structures is degraded at first (Higuchi 1985).

The highest Klason lignin was found in the media made of gamal wood (28.56%) and it was statistically different with the media made of other wood species. Przybylowicz and Donoghue (1990) pointed out that lignin has special role in Shiitake mushroom growth. Shiitake mushroom also obtains the nutritions needed from lignin degradation process result. Shiitake mushroom's hypha extracts enzymes which are able to destroy the undegradable materials such as cellulose and lignin from the wood and change them into the simple monomers then give back to the hypha of fungi as food. Meanwhile, no significant difference was found in the acid soluble lignin content among the wood species, statistically.

Table 2. Chemical contents of media from 4 wood species (%).

Chemical content	Sengon	Gamal	Johar	Lamtoro	ANOVA Analysis
Ethanol-toluene extractives	4.45±0.02^{a}	8.36±0.11^{c}	8.84±0.04^{d}	5.73±0.12^{b}	**
Hot water soluble extractives	5.06±0.16^{b}	11.42±0.13^{c}	16.3±0.19^{d}	2.95±0.06^{a}	**
Holocellulose	81.59±0.70^{c}	75.85±0.13^{b}	70.33±0.37^{a}	84.67±0.99^{d}	**
α-cellulose	52.86±0.25^{a}	58.97±0.50^{b}	51.63±0.76^{a}	60.04±0.64^{b}	**
Hemicellulose	28.73±0.78^{c}	16.89±0.48^{a}	18.7±0.84^{a}	24.63±1.61^{b}	**
Klason lignin	22.15±1.00^{a}	28.56±0.30^{b}	23.77±0.14^{a}	22.32±0.96^{a}	**
Acid soluble lignin	0.03±0.002	0.03±0.001	0.03±0.004	0.03±0.001	ns

The same superscript letter followed by the average value in the same row shows no significant difference among the four tree species by Tukey-Kramer test at the 5% level. n = 3. ± = Standard deviation.

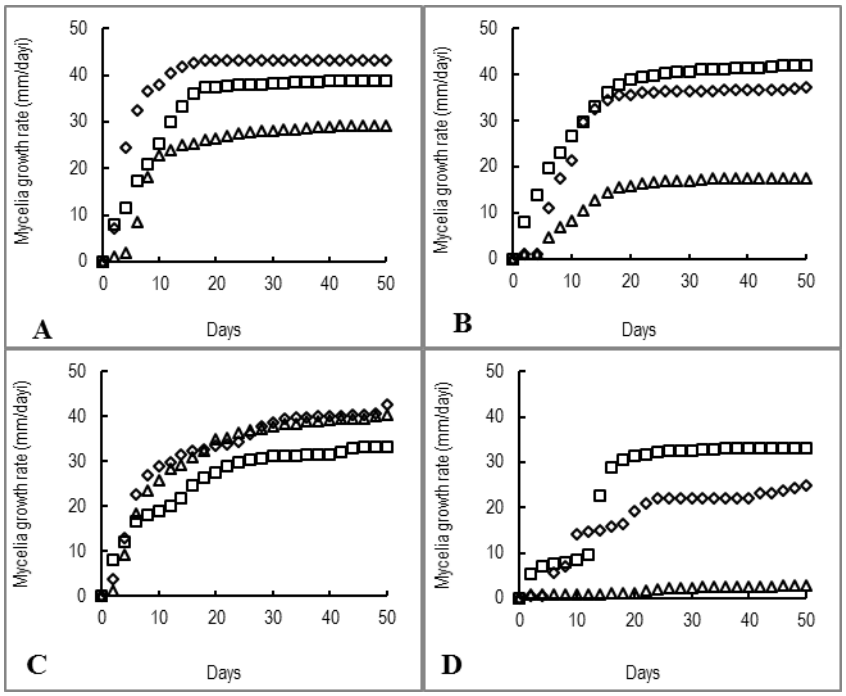

Figure 1. Graphic of Shiitake mushroom's mycellia growth. Remarks: (A) Gamal; (B) Johar; (C) Lamtoro; (D) Sengon. Diamond (65%); Square (70%); Triangle (75%).

Mycellia Growth Rate

Mycellia growth rates per 2 days on each types of substrate with different moisture content were shown in Fig. 1. Those 4 graphics showed that the average length of mycellia growth rate increased rapidly on the beginning of growth period and then it remained constant relatively. The average value of mycellia growth rate on this research was ranged from 0.61 to 3.32 mm/day. This value is included in the range of another mushroom growth rate which is *Auricularia polytrica*, which has growth rate among 1.80~3.21 mm/day (Irawati *et al*. 2012). Statistic analysis results of mycellia growth rate on factor wood species and moisture content showed no significant effect on factor wood species and the interaction of those 2 factors, however, factor moisture content alone very significantly affected to mycellia growth rate.

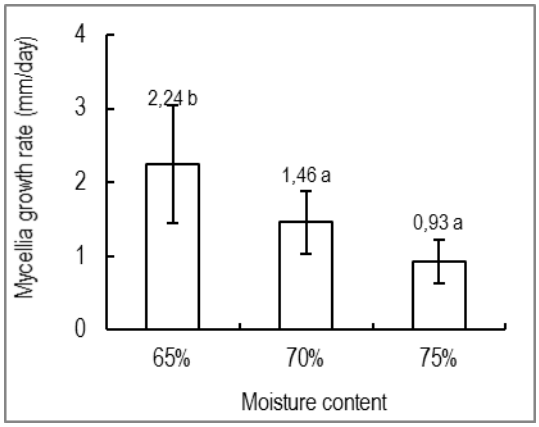

Figure 2. Graphic of mycellia growth in 3 different moisture contents.

Fig. 2 shows the values of mycellia growth rate on 65% moisture content is significantly different with mycellia growth rate on 70% and 75% moisture content. Whereas no significant difference was found on the values of mycellia growth rate on 70% moisture content and 75% moisture content, statistically. The highest mycellia growth rate value was found media with 65% moisture content which is 2.24 mm/day. Actually, according to the previous research results, Shiitake is classified in mushrooms with high water activity, means Shiitake mushroom will grow well on media which has high moisture content (Hu *et al*. 2004), however it also pointed out that water activity of Shiitake mushroom is also affected wood physical properties as media. Wood with high water activity (easy to absorp water) needs high water addition in order to be good as Shiitake mushroom cultivation media, and vice versa. In this research, the woods used were supposed as species with low water activity, so the optimum water addition was 65%. Widyastuti (2009) pointed out that moisture content of sawdust media for Shiitake mushroom vegetative growth is ranged from 60 to 75%. Mycellia growth rate of Shiitake on media made of sugi sawdust increases along with the increment of moisture content of media from 65 to 75%, it was also happenned on media made of konara wood (Hu *et al*. 2004).

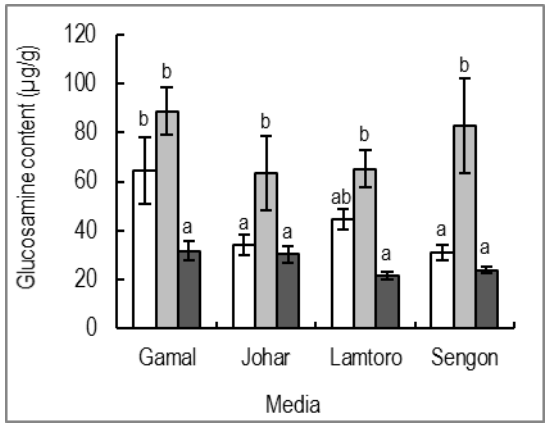

Figure 3. Graphic of glucosamine content of Shiitake mushroom's mycellia on 4 types of media. White (65%). Light gray (70%). Dark gray (75%).

Glucosamine Content

Glucosamine content was measured to determine the number of mycellia on media substrate before fruiting bodies phase. No branches polymer from N-acetyl-D-glucosamine (chitin) is component which is avalilable on mushroom's cell wall, generally. Acid hydrolysis to kitin will disintegrate glucosamine and comparison between glucosamine and mycellia dry-weight can be determined and used to calculate mushroom biomass in the media or substrate (Jones and Worral 1995). Ohga (2000) pointed out that the bigger glucosamine content makes the number of mycellia in the media substrate more abundant.

The average value of glucosamine content in this research was ranged among 21.56~88.81 µg. Fig. 3 showed that media glucosamine content made of 4 wood species with 70% moisture content posses highest value compared to media with different moisture content indicating that 70% moisture content is the most suitable moisture content for Shiitake mushroom's mycellia growth on 4 media. Media moisture content affects to mushroom growth, because ideal moisture content in the baglog will yield which

is not too wet or dry. Too high moisture content in the baglog may cause that baglog will be decomposed quickly, in contrary, less moisture content in the baglog also causes not optimum mushroom growth (Meinanda 2013).

Correlation between Chemical Content to Growth Rate and Glucosamine Content

Wood mushrooms, in their growth period, generally obtain the nutritions from the wood decomposition of their media. The main components of wood composer which are cellulose, hemicellulose and lignin are carbon resources which are needed for wood mushroom's growth (Przybylowicz and Donoghue 1990). However, Przybylowicz and Donoghue (1990) also added that resins and phenolic compounds may inhibit the mushroom growth on wood media both hardwood or softwood because those kind of extractives are very difficult to be degraded by wood mushroom, generally.

Correlation analysis results between chemical content of sawdust media and mycellia growth rate and glucosamine content are presented in Table 3. The average of Shiitake mycellia growth rate in this research positively correlated with ethanol-toluene extractives content and negatively correlated with hemicellulose content of the media. This results indicated that media with high ethanol-toluene extractives content can be grown by mushroom mycellia faster. Whereas on the media have high hemicellulose content mushroom mycellia grew slower. Generally, extractives content (especially ethanol-toluene extractives content) may cause negative impact to mycellia growth (Przybylowicz and Donoghue 1990), however, Shiitake mushroom in this research used substrates in ethanol-toluene extractives as carbon resources for its growth. Shiitake mushroom is classified as white-rot fungi therefore Shiitake mushroom secretes ligninase enzyme to degrade lignin or other phenolic compounds in the media. At first, Shiitake mushroom is supposed to use the simple compounds in the extractives and then degrade lignin which is more complex. This matter also explains about the high correations between lignin content and growth rate or glucosamine content, however, statistically those correlation coefficients are not significant. Negative correlation between mycellia growth and hemicellulose content pointed out that Shiitake mushroom does not use hemicellulose of the media for its growth. The same tendency was also happenned on holocellulose although the correlation coefficient was not significant. Shiitake mushroom does not secrete hemicellulose enzyme to degrade cellulose, allegedly.

Table 3. Correlation of chemical content to growth rate and glucosamine content.

Chemical components	Growth rate	Glucosamine content
Ethanol – toluene extractives	0.973 *	0.350 ns
Hot water soluble extractives	0.853 ns	0.169 ns
Holocellulose	-0.821 ns	-0.141 ns
α-cellulose	0.042 ns	0.476 ns
Hemicellulose	-0.988 *	-0.534 ns
Klason lignin	0.825 ns	0.927 ns

** = Significant at 1% level. ns = Not significant.

Conclusions

The growth rate of mycelia was affected by the moisture content of media, meanwhile the glucosamin content is influenced by the moisture content and various species of wood. The highest glucosamine content was found in 70% moisture content for all species, although the highest mycelia growth rate was found in 65% moisture content. The best combination to cultivate the Shiitake mushroom was by using media made of gamal with 70% moisture content. There were positive correlation among the ethanol-toluen extracts content and the mycelia growth, however there was negative correlation among the hemicelullose content the mycelia growth.

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WOOD RESEARCH Journal

Journal of Indonesian Wood Research Society

Annals of the Wood Research Journal

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Conclusions (and Suggestions)
References
Name and complete address of Authors
Appendix

4. Other rules:

4.1. Names of wood are followed by Botanical Name.
4.2. Values between are written using this symbol (~), e.g. 3.75 ~ 8.92%.
4.3. Editors could modify Figures without changing their substantial meaning.
4.4. References are arranged from A to Z.
4.5. References in text are written as this example: (Palomar *et al.* 1990; Arancon 1997).
4.6. Examples of writing of References: Altschul, S.F.; T.L. Madden; A.A. Schäffer; J. Zhang; Z. Zhang; W. Miller; D.J. Lipman. 1997. Gapped BLAST and PSI-BLAST: A New Generation of Protein Database Search Programs. Nucleic Acids Res. 25: 3389-3402.

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Example of Table and Figure

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

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

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

Source: Chujo *et al.* 2010.

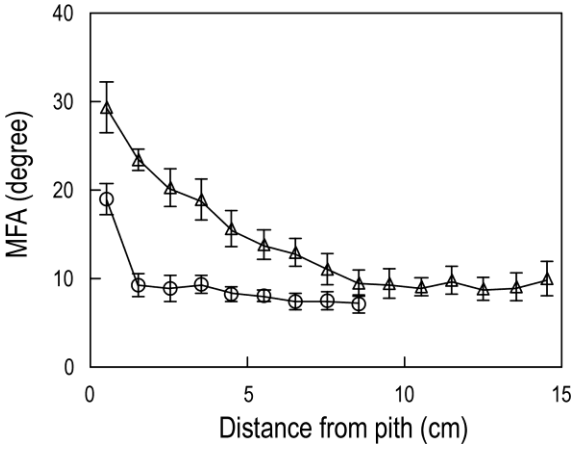

Figure 3. Radial variation of microfibril angle of the S2 layer in tracheid. Open circle, *Agathis* sp.; open triangle, *Pinus insularis*; Bars indicate the standard deviation. (Source: Ishiguri *et al.* 2010)


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