# CHARACTERISTICS OF AEROGEL MADE FROM BALSA AND PULAI WOOD

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## *ABSTRACT*

The greenhouse effect which has a negative impact on human life has encouraged the production of a material that can capture and store carbon, called aerogel. Unfortunately, this aerogel is made using porous metal as raw material, which is quite expensive to procure. This research aims to examine the effect of a two-stage delignification process on pore diameter and porosity, wood color and density as well as weight loss of wood aerogel made of balsa and pulai wood, which come from community gardens around the IPB Darmaga campus, Bogor. The first delignification process was carried out using $Na_2SO_3$+NaOH solution for 8, 9 and 10 hours at a temperature of 100 ºC and then followed by using $H_2O_2$ for 1 and 3 hours at the same temperature. Before testing, the delignified samples were frozen at –20 ºC for 24 hours then freeze-dried at –50 ºC for 48 hours and air-dried. The results show that the two-stage delignification process does not affect the types of cells wood, but causes the pore diameter to increase, the wood color becomes brighter and the wood becomes more porous, while the density and weight decreases. This is related to the degradation of lignin and hemicellulose components. In general, the best treatment is 9N1H, namely a combination of delignification of $Na_2SO_3$+NaOH for 9 hours and $H_2O_2$ for 1 hour at a temperature of 100 ºC. The results also show that pulai wood is more resistant to two-stage delignification than balsa wood.



*Keywords: Aerogel, balsa, delignification, greenhouse effect, pulai*


## INTRODUCTION

Global warming is a serious problem that must be anticipated immediately because it can cause disaster for all living things. According to Purnamasari (2019), greenhouse gases such as $CO_2$, $CH_4$ and $N_2O$ are the main causal factors. Global warming will result in climate change, which will then have a negative impact on ecosystems, food security, water availability and human health. In the context of climate change, the forestry sector must be responsible because several activities in the forestry sector such as forest fires, changes in the function and use of land and forest areas, as well as the use of energy from organic materials have negative impacts (Maryani 2020). According to Wibowo (2013), Indonesia's forestry sector contributes around 48% of total carbon emissions, and $CO_2$ is the largest contributor (± 77% of total emissions). Therefore, success in capturing and storing $CO_2$ will be able to reduce the rate of climate change and global warming. This requires a material that is able to capture and absorb carbon, known as aerogel.

Aerogel is a super light material with a very limited solid phase, very elastic, low conductivity, resistant to heat and also functions as a place for nanoparticle deposition (Gustinenda and Margo 2017). According to Roy et al. (2023), aerogel also has the potential to be an efficient and environmentally friendly thermal insulation. So far, the raw materials for making aerogel are porous metals such as silica, aluminum, zeolite and graphene. Procurement of these materials requires relatively high costs (Mai Bui et al. 2018). Therefore, it is necessary to look for other materials as raw material that are cheaper, but are still capable of capturing and storing carbon while helping industrial decarbonization and reducing $CO_2$ emissions into the atmosphere. One of the porous solid adsorbents that has the potential to be developed is wood because it naturally has cavities, abundant, environmentally friendly, easily degraded organically and has good mechanical properties and does not require special handling (Fitria 2008; Weinheim et al. 2022). According to Berglund and Burgert (2018), with an organized molecular structure–consisting of cellulose, hemicellulose and lignin–which are interconnected to form an open lumen as well as a natural hierarchical arrangement and better composition, wood has the potential to be used as a raw material for making aerogel.

The process of making aerogel always begins with the removal of lignin (delignification). The resulting intermediate product is called the forerunner or precursor of aerogel. So far, the characteristics of aerogel precursors have never been studied. In fact, by knowing the characteristics of the aerogel precursor, the best delignification method can be found. Indonesian tropical wood species have never been used as raw material. Apart from being expensive, the characteristics of metal aerogels are still not optimal (Mai Bui et al. 2018). The delignification process carried out is generally only one stage.

Considering these things, therefore, this research was carried out in order to find the best delignification method for two species of Indonesian tropical wood, namely balsa (*Ochroma bicolor*) and pulai (*Alstonia scholaris*) wood. The main focus is to study changes in wood characteristics after the wood has been delignified in two-stage but has not been impregnated with resin and polymerized with methyl methacrylate. Because the solvents and period time of delignification process are determining factors, the combination of these two factors will be analyzed. The first delignification was carried out using $Na_2SO_3$+NaOH solution for 8, 9 and 10 hours, while the second delignification used $H_2O_2$ solution for 1 and 3 hours. The temperature used is 100 ºC. After delignification, the samples were frozen at –20 ºC for 24 hours and then freeze-dried at –50 ºC for 48 hours. This double-stage delignification is believed to be able to remove more lignin thereby increasing the ability of aerogel to capture and store carbon.

The aim of this research was to examine the effect of the two-stage delignification process on the anatomical structure, especially pore diameter and cell cavity portion (porosity), wood color and density as well as the weight loss in order to find the optimal solvent and duration for the delignification process for making aerogel from balsa and pulai wood.

## METHODOLOGY

### Main Material

The main materials used are balsa (*O. bicolor*) wood, 25 cm in diameter, and pulai (*A. scholaris*) wood, 20 cm in diameter. Both species are round log with a length of ± 100 cm. Other supporting materials are sodium sulfite ($Na_2SO_3$), sodium hydroxide (NaOH), peroxide ($H_2O_2$) and distilled water. Balsa and pulai wood were obtained from community forests around the IPB campus, while chemicals were purchased from Sigma Agric. The equipment used consists of a light microscope, USB integrated with a computer, microtome, oven, magnetic hot plate, beaker glass, digital analytic balance, caliper, measuring glass, thermometer and refrigerator.

### Research Procedure

The research procedure consisted of sample and solvent preparation, a two-stage delignification process, sample freezing and freeze-drying processes, physical property testing, anatomical structures and color changes measurement and measuring the weight loss. The research flow diagram before testing is presented in Figure 1.

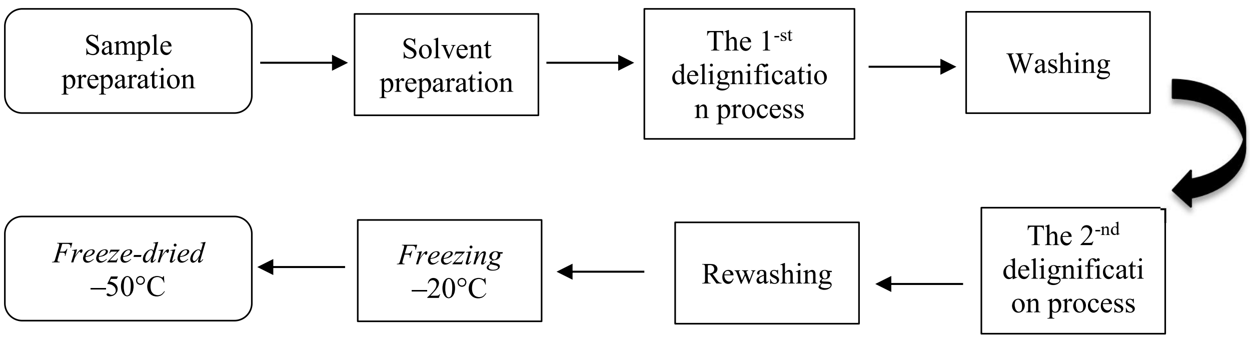


Figure 1. Research flow diagram before testing

### Sample Preparation

The balsa and pulai logs were converted into disks and planks, then reconverted into square logs and then air-dried until a sample with a final size of 2 × 2 × 1 mm was obtained. The number of samples per species and per treatment was 10 replications. The code and description of each treatment per wood species are presented in Table 1.

Table 1 Sample code, size and description of each treatment per species

| Species | Code | Delignification Treatment | Sample Size (mm) |
|---|---|---|---|
| Balsa | BFD | Control | 2 × 2 × 1 |
| | B8N1H | In $Na_2SO_3$+NaOH 8 hrs. and then in $H_2O_2$ 1 hr. | |
| | B8N3H | In $Na_2SO_3$+NaOH 8 hrs. and then in $H_2O_2$ 3 hrs. | |
| | B9N1H | In $Na_2SO_3$+NaOH 9 hrs. and then in $H_2O_2$ 1 hr. | |
| | B9N3H | In $Na_2SO_3$+NaOH 9 hrs. and then in $H_2O_2$ 3 hrs. | |
| | B10N1H | In $Na_2SO_3$+NaOH 10 hrs. and then in $H_2O_2$ 1 hr. | |
| | B10N3H | In $Na_2SO_3$+NaOH 10 hrs. and then in $H_2O_2$ 3 hrs. | |
| Pulai | PFD | Control | 2 × 2 × 1 |
| | P8N1H | In $Na_2SO_3$+NaOH 8 hrs. and then in $H_2O_2$ 1 hr. | |
| | P8N3H | In $Na_2SO_3$+NaOH 8 hrs. and then in $H_2O_2$ 3 hrs. | |
| | P9N1H | In $Na_2SO_3$+NaOH 9 hrs. and then in $H_2O_2$ 1 hr. | |
| | P9N3H | In $Na_2SO_3$+NaOH 9 hrs. and then in $H_2O_2$ 3 hrs. | |
| | P10N1H | In $Na_2SO_3$+NaOH 10 hrs. and then in $H_2O_2$ 1 hr. | |
| | P10N3H | In $Na_2SO_3$+NaOH 10 hrs. and then in $H_2O_2$ 3 hrs. | |

**Solvent Preparation**

The first solvent is a mixture of $Na_2SO_3$+NaOH (1:1 v/v) with a pH of 4.6; while the second one is a mixture of $H_2O_2$ and distilled water with a concentration of 6%. Each solution was prepared until mixed perfectly.

**Delignification Process**

Delignification is carried out at two stages. It begins by boiling the sample in a beaker glass containing $Na_2SO_3$+NaOH solution for 8, 9 and 10 hours at 100 °C, then washing it with distilled water 3 times @ 1 minute until it is free of alkali, and then continuing by boiling the sample in $H_2O_2$ solution for 1 and 3 hours at 100 °C, then washed again with distilled water several times until the pH of the solution is neutral. The samples were then frozen in a freezer (–20 °C) for 24 hours then freeze-dried in a freeze drier (–50 °C) for 48 hours and ready to be observed for the anatomical structure and wood color as well as measuring the density, pore diameter, porosity and weight loss.

**Anatomical Structure and Wood Color Observation**

The anatomical structure was observed directly using a microscope equipped with a USB connected to a computer with a magnification of 100×, while the color of wood was determined using a color meter. The surface observed is the cross section. Apart from the organization of the cells that make up the wood, the pore diameter and percentage or portion of cell cavities, namely porosity, were also observed and measured and then averaged with the help of image-J software. The same parameters were also carried out for control wood. Pore diameter is the average value of 50 intact individual cells. The pore diameter and porosity were also calculated with the help of image-J software. The porosity is determined using the following formula:

$$\text{Porosity (\%)} = \frac{Area\ of\ cell\ cavity}{Total\ area} \times 100$$

The wood color is determined by Lab and Lch parameters. Lab represents an integrated color where a represents a reddish green color and b represents a bluish yellow color; while Lch represents the brightness and saturation of the color. The color change is calculated based on the following equation:

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

$$\Delta E* = \sqrt{\Delta L^2 + \Delta c^2 + \Delta h^2}$$

Where:
$\Delta L$ = Difference of $L_2$ (before treatment) and $L_1$ (after treatment)
$\Delta a*$ = Difference of a* before and after treatment
$\Delta b*$ = Difference of b* before and after treatment
$\Delta c*$ = Difference of c* before and after treatment
$\Delta h*$ = Difference of h* before and after treatment
$\Delta E*$ = Color changes before and after treatment

**Wood Density, Pore Diameter and Porosity Measurement**

The density of control wood and delignified wood (aerogel precursor) was measured gravimetrically. The value is determined based on the comparison between the weight and volume under the same conditions; and expressed in g/cm$^3$. The density of wood is determined by the formula:

$$\text{Density (g/cm}^3\text{)} = \frac{BA}{VA}$$

Where:
BA = Sample weight in air-dried condition (g)
VA = Sample volume in air-dried condition (cm$^3$)

**Weight-loss Measurement**

Weight loss in the delignification process is to evaluate the level of efficiency and success of the lignin removal process. Weight loss was calculated by the formula:

$$\text{Weight loss (\%)} = \frac{BA - BB}{BA} \times 100$$

Where:
BA = Sample weight before treatment (g)
BB = Sample weight after treatment (g)

**Data Analysis**

Qualitative data is presented in the form of photographs and described narratively, while quantitative data is calculated by average values and standard deviations and presented in table or graphs. Changes in anatomical structure and physical properties, color and pore diameter due to the delignification process are discussed comprehensively. The effect of the delignification process on the parameters studied was statistically analyzed using ANOVA and continued with the Duncan test.

## RESULTS AND DISCUSSION

**Anatomical Structure and Wood Color**

The results of the anatomical structure observations are presented in Figures 2 and 3, pore diameter in Table 2, while changes in wood color are in Figures 4 and 5. It is known that the double stage delignification process does not result in changes to the appearance of wood, but does result in an increase in the size of the pore diameter of both species (Table 2). The cross-section appearance of delignified balsa and pulai wood is still the same as the appearance of the control wood, except for the size of the pore diameter. Compared to the control, the pore diameter of balsa wood increased by 4.07–34.93% with an average of 23.67%; while the diameter of pulai wood increased 0.90–32.26% with an average of 19.56%. Overall, the increase in pore diameter of balsa wood after delignification was higher than that of pulai.

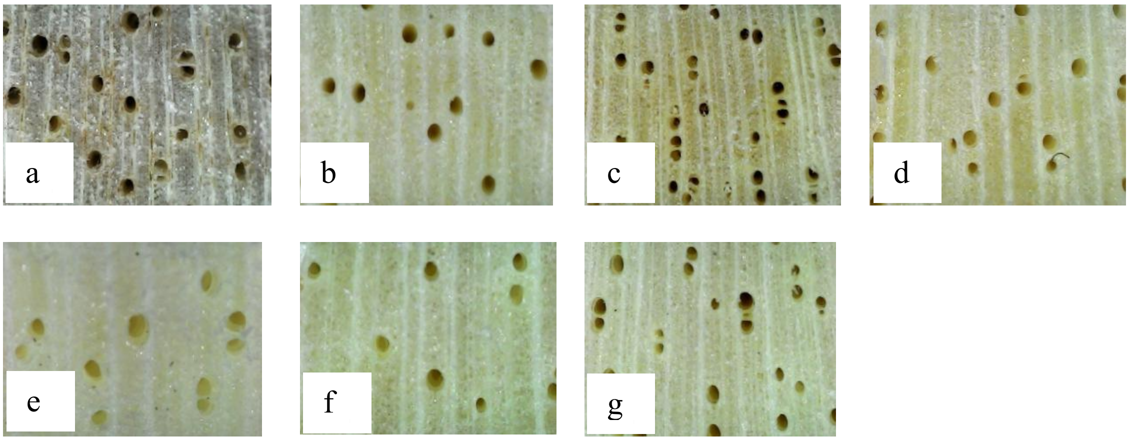

Figure 2 Cross-section of Balsa wood: a) control, b) 8N1H, c) 8N3H, d) 9N1H, e) 9N3H, f) 10N1H and g) 10N3H

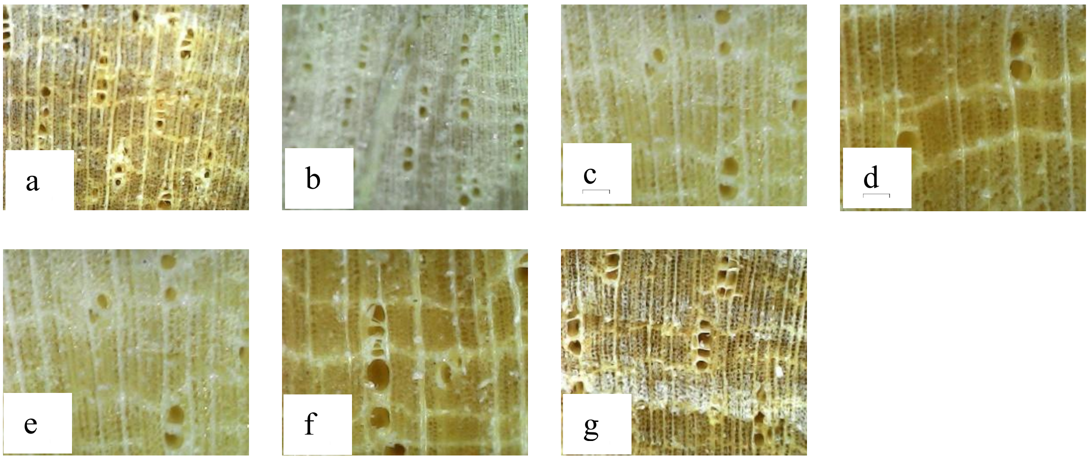

Figure 3 Cross-section of Pulai wood: a) control, b) 8N1H, c) 8N3H, d) 9N1H, e) 9N3H, f) 10N1H and g) 10N3H

From the two pictures above, it is known that the characteristics of balsa wood are as follows: the porosity is diffuse porous, solitary pores are dominant even though there are radial multiple 2 pores, ray parenchyma is clear but axial parenchyma is not visible. Meanwhile, the structural characteristic of Pulai wood is diffuse porous, radial multiple 2–4 pores are dominant, although there are also solitary ones, the ray and axial parenchyma are clear. The structural characteristics of both wood species are similar to the original wood (Ogata et al. 2018). There were no differences indicating that the delignification process had no impact on the anatomical structural characteristics of the wood.

The results of this study are also in accordance with Zhu et al. (2019) which states that after lignin and hemicellulose are removed, the aerogel precursor will still maintain the existence of its constituent cells because of the strong and complex cellulose framework. In both figures it can also be seen that the aerogel precursor still has pores like the control wood but the diameter tends to increase (Table 2). This is in accordance with Zhuo et al. (2016). The measurement results also show that the average pore diameter increases with increasing delignification time. This indicates that process of removing lignin and hemicellulose during delignification tends to increase the size of wood pores.

Table 2 Average pore diameter in balsa and pulai wood in each treatment

| Sample/Treatment Code | Pore Diameter of Balsa (μm) | Pore Diameter of Pulai (μm) |
|---|---|---|
| FD | 277.4 (7.9) | 266.0 (5.6) |
| 8N1H | 288.7 (7.9) | 268.4 (8.0) |
| 8N3H | 309.9 (8.8) | 305.3 (9.7) |
| 9N1H | 347.1 (9.5) | 316.9 (10.6) |
| 9N3H | 367.6 (9.0) | 322.2 (10.1) |
| 10N1H | 370.7 (6.3) | 343.6 (11.2) |
| 10N3H | 374.3 (8.4) | 351.8 (14.2) |

Note: The numbers in brackets represent the standard deviation values

In terms of treatment, the combination of $Na_2SO_3$+NaOH for 10 hours and $H_2O_2$ for 3 hours (10N3H) resulted in the highest increase in pore diameter size in both species, while the 8N1H treatment (combination of $Na_2SO_3$+NaOH 8 hours and $H_2O_2$ 1 hour) was the lowest. In terms of species, balsa wood has a larger pore diameter increase compared to pulai wood. This is related to wood density where denser wood has higher lignin contents (Nawawi et al. 2019). These two findings indicate that the increase in pore diameter size is more dependent on the delignification time and wood species. The longer the delignification time and the denser the wood, the pore diameter tends to increase. This is in line with Rollison (2003); Chmiola et al. (2006) where delignification can open porous networks, change the size of macro pores (> 50 nanometers) and increase the size of micro pores. And as a result, the aerogel precursor has a low density and conductivity, but its surface area increases.

Figures 4 and 5 show the change in wood color. The color of wood tends to become lighter as the delignification time increases. The increase in color brightness is thought to be related to the degradation of lignin and hemicellulose.

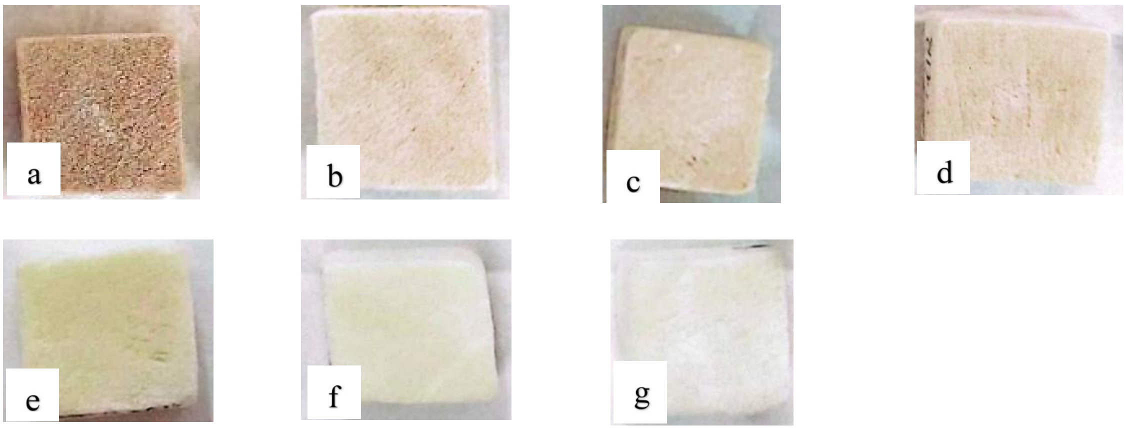


Figure 4 Color change in Balsa wood: a) control, b) 8N1H, c) 8N3H, d) 9N1H, e) 9N3H, f) 10N1H and g) 10N3H

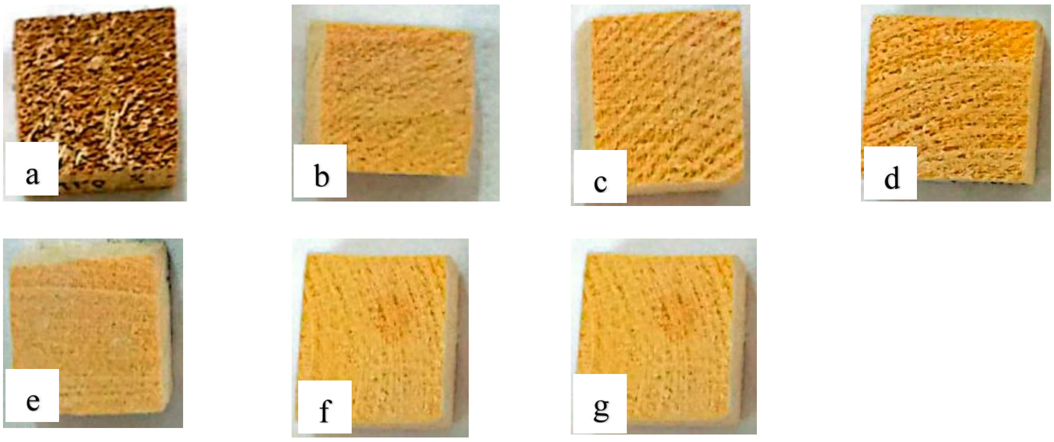


Figure 5 Color change in Pulai wood: a) control, b) 8N1H, c) 8N3H, d) 9N1H, e) 9N3H, f) 10N1H and g) 10N3H

The color change of wood after double stage delignification which includes ΔE Lab, ΔE Lch and L is presented in Figure 6. The ΔE Lab value in the context of color testing refers to the color difference using certain methods and devices. Delta (Δ) indicates the difference or change in color that occurs. The Lab color coordinate system is used to describe color with L for brightness, a for the position between green and red and b for the position between blue and yellow.

From Figure 6, it is known that the ΔE Lab value of the resulting aerogel precursor increased compared to the control. The color of the aerogel precursor for both species was brighter than the control wood. The aerogel precursor made of balsa wood was brighter than the pulai wood aerogel precursor. It is also seen that the brightness level of wood color increases with increasing delignification time. Likewise, with the ΔE Lch value (Figure 6-middle) and the L value (Figure 6-bottom). Overall, it can be said that the balsa wood aerogel precursor is brighter than pulai wood except for the 8N3H treatment. These findings indicate that there is an influence of delignification on wood color.

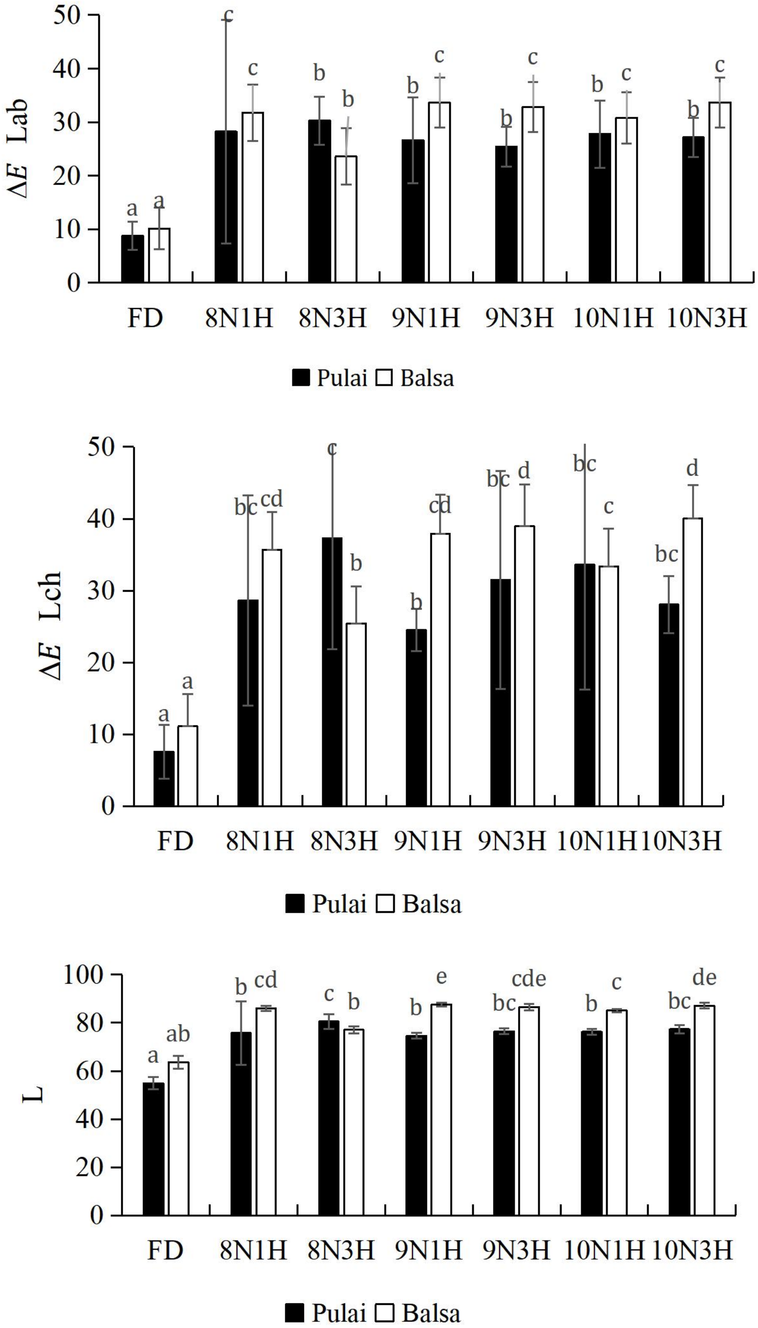


Figure 6 Change in wood color: ΔE Lab (above), ΔE Lch (middle) and L (bottom)

The research results show that the 9N1H is the best treatment for balsa wood in terms of brightness level, while the best treatment for pulai wood is the 8N3H. This is supported by Figure 7 for balsa wood as an example and Table 3.

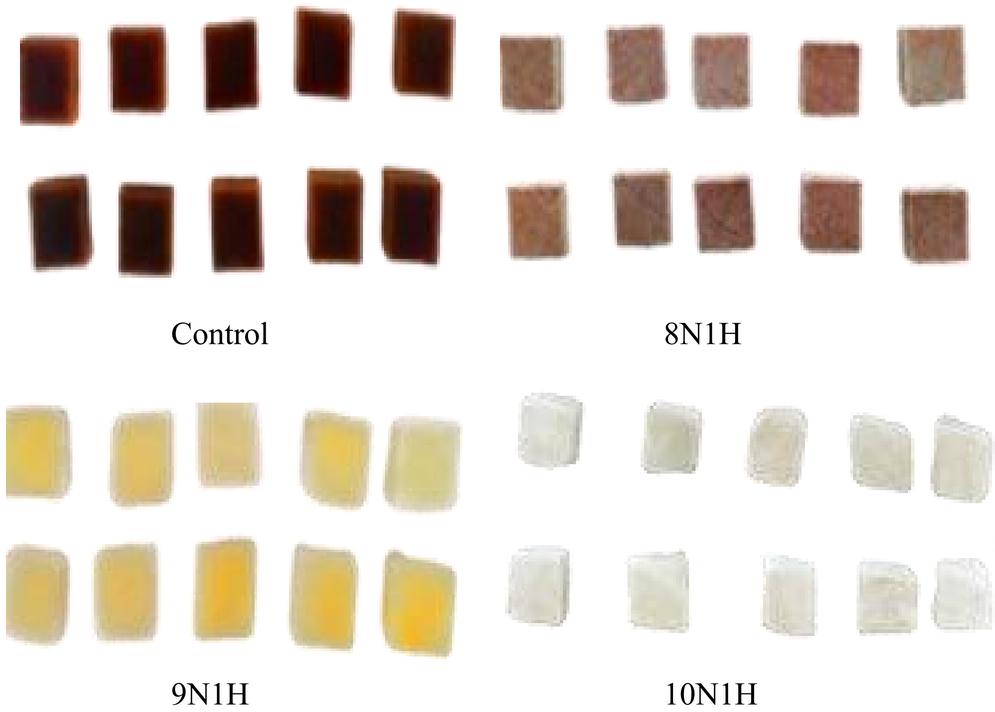


Figure 7 Changes in color of Balsa wood due to double stage delignification

Table 3 Wood color based on *database*

| | BALSA | | | PULAI | | |
|---|---|---|---|---|---|---|
| FD | AF9674 | Tan | | A27B44 | Peru | |
| 8N1H | E3D6B0 | Wheat | | DBB866 | Dark Khaki | |
| 8N3H | D1BD89 | Tan | | DDC592 | Burlywood | |
| 9N1H | E8DDB0 | Gold | | E1B066 | Sandy Brown | |
| 9N3H | DED7BA | Gold | | D5B476 | Goldenrod | |
| 10N1H | E4D1AA | Gold | | E6B76B | Burlywood | |
| 10N3H | DDD6C0 | Antique White | | D9BB87 | Burlywood | |

**Wood Density and Porosity**

The calculation results show that the density of balsa and pulai wood after double-stage delignification tends to be lower than control wood. This value tends to decrease with increasing delignification time (Figure 8). Compared to the control, the density of balsa wood after double-stage delignification was reduced by 22.43–34.68% with an average of 25.97%; while the density of pulai wood decreased by 16.48–25.58% with an average of 17.98%. In general, the decrease in the density of balsa wood after delignification is higher than that of pulai wood (Figure 8). These findings indicate that double-stage delignification is able to reduce wood density. This is related to the degradation of lignin and hemicellulose components. The treatment that effectively reduces the density of wood is the 8N3H for both species.

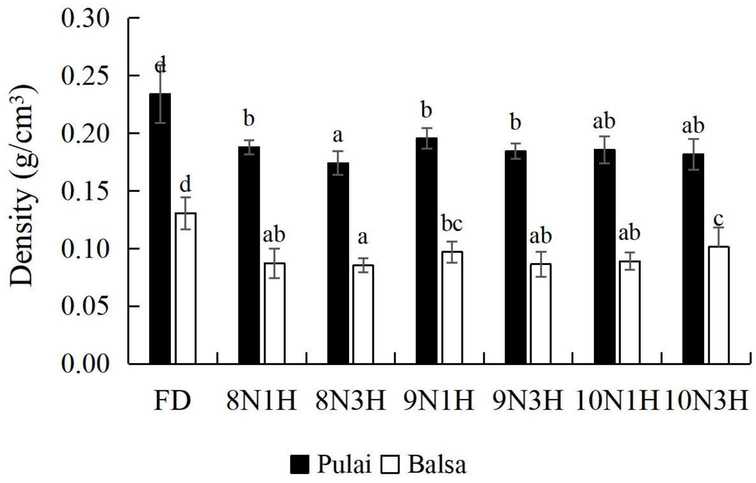


Figure 8 Comparison of wood density value

The reduction in wood density is supported by the increase in wood porosity. The research results (Figure 9) support this opinion. Compared to the control, the porosity of balsa wood after delignification increased by 108.47–251.42% with an average of 164.16%; while the porosity of pulai wood increased 9.94–33.85% with an average of 22.94%. Increasing porosity is positively correlated with increasing delignification time and is related to reducing wood density values and increasing pore diameter. The appearance of micro-voids in the cell walls and damage to the middle lamella due to degradation of lignin and hemicellulose components also increase wood porosity. This is in accordance with Song et al. (2017); Sun et al. (2020); Adkhamjon et al. (2021). Overall, the treatment that effectively increases wood porosity is the 10N3H for balsa, while for pulai it is 9N3H.

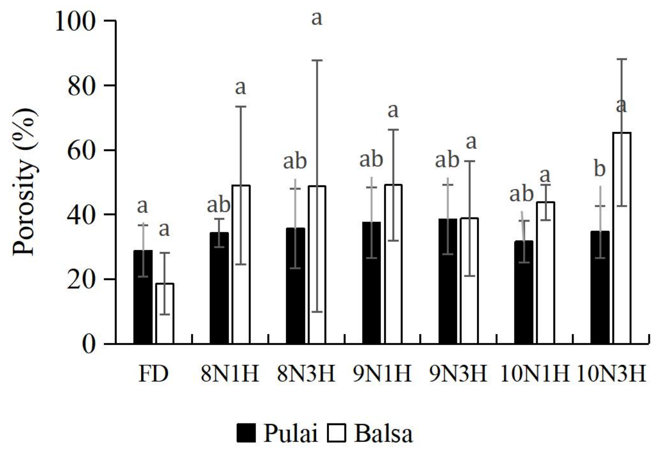


Figure 9 Changes in porosity due to double-stage delignification

***Weight Loss***

The results show that weight loss due to double-stage delignification is influenced by the wood species. The weight loss is relatively constant although it fluctuates with increasing delignification time (Figure 10). Weight loss in balsa wood ranges from 25.48–37.30% with an average of 31.09%; while for pulai wood it is 14.34–20.24% with an average of 16.91%. The reduction in wood weight is also related to the degradation of lignin and hemicellulose components. This is in accordance with Septevani et al. (2018). Overall, the best treatment for balsa wood is 10N1H, while for pulai wood it is 9N3H.

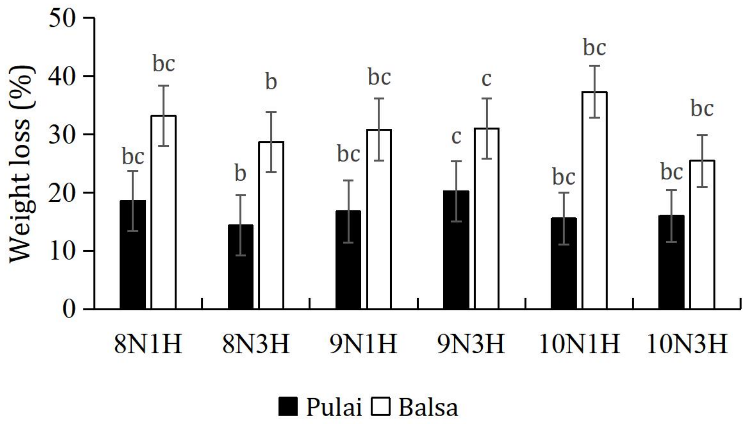


Figure 10 Weight loss due to double-stage delignification

The lower weight loss of pulai wood compared to the weight loss of balsa wood indicates that pulai wood is more resistant to the delignification process. In other words, the lignin in pulai wood is more difficult to dissolve. The use of NaOH not only affects lignin, but also hemicellulose and cellulose. Partial degradation and loss of acetyl groups and uronic acid in hemicellulose causes a reduction in hemicellulose content; whereas for cellulose, the use of NaOH causes the cellulose to expand and its crystallinity to decrease (Xu and Huang 2014: Harmsen et al. 2010; Wu and Jahim 2016).

## CONCLUSION

Double-stage delignification does not affect the types of cells that make up the wood, but causes the pore diameter to increase, the wood becomes brighter and more porous, while the density and weight of the wood decreases. These changes become more significant with increasing delignification time. An effective delignification treatment for both types of wood is 9N1H.

In general, pulai wood is more resistant to applied double-stage delignification than balsa wood.

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