Physical properties of impregnated sengon wood by monoethylen glycol and nano silica from betung bamboo sticks

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Abstract. Sengon wood (Falcataria moluccana) is a fast-growing species that has low dimensional stability and density. The purposes of this study were to analyze the effect of impregnation treatment by using MEG (monoethylen glycol) and nano silica from betung bamboo sticks on the dimensional stability and density of sengon wood and to identify the characteristics of impregnated sengon wood. The treatments were consisted of untreated (water treated), 50% MEG (MEG), 0.5% nano silica in 50% MEG (MEGsilica 0.5%), 0.75% nano silica in 50% MEG (MEGsilica 0.75%) and 1% nano silica in 50% MEG (MEGsilica 1%). Weight percent gain, anti-swelling efficiency, water uptake, bulking effect, and density were affected by the treatments. It was due to the coverage of MEG and nano silica on cell walls and vessels of sengon wood (SEM analysis). Also, the presence of nano silica in wood treated (SEM-EDX analysis). The cristalinity of sengon wood decreases because MEG is amorphous. FT-IR showed there was no MEG bonds with sengon wood cell wall components or between nano silica and sengon wood. The optimum treatment for sengon wood was MEGsilika 1%.

1. Introduction

Wood is one of the most popular raw materials for various needs in Indonesia. Every year wood production in Indonesia continues to decline with increasing demand. The need for wood in Indonesia was 10,300,872 m³, but natural forests were only able to provide 5,843,179 m³ [1]. Therefore, plantation and community forest are useful in supplying timber for the timber industry in Indonesia. Fast growing species were widely planted in plantation and community forest

Fast growing wood has inferior characteristics i.e. low strength and density and high portion of juvenile wood compared to slow growing wood. Juvenile wood has lower physical, mechanical strength and biodeterioration resistance compared to mature wood [2]. One of the fast-growing woods is sengon. Sengon wood has an average specific gravity of 0.3-0.5, durable class IV-V and strong class IV-V [3]. Therefore, wood modification is an alternative to improve wood quality.

Some of the basic properties of wood in the form of durability, dimensional stability, and hardness of wood can be improved by wood modification techniques [4]. Some wood modification treatments include chemical modification, thermal modification, surface modification and impregnation method. Impregnation is performed by introducing chemicals into the wood cell walls in order to increase the characteristics of wood [5].
The impregnation process is a method to alter wood properties by intervening at cell wall level. The chemicals that are commonly used include styrene [6] and methyl metacrylate (MMA) [7]. Impregnation can keep and precipitate chemicals into cells without damaging the wood so that the wood becomes more dense [8], thus it can overcome the problem of dimensional stability and density of wood. [9] impregnated sengon wood using MEG (Monoethylen glycol) and nano SiO$_2$. The results showed that dimensional stability and density of sengon wood increase because MEG and nano SiO$_2$ can easily enter the pits and wood cell walls.

Silica is a material that usually used for thermal insulators, catalyst supports, adsorbents, drug delivery, glass fillers, glass, and raw materials for making solar cells [10]. Silica sources can be found in inorganic materials such as sand, soil, rocks [11], as well as in organic materials known as bio-silica such as [12] rice husks and betung bamboo [13]. Utilization of silica can be expanded by synthesizing in micro or nano size. Micro-sized silica can improve the dimensional stability of materials, according to [14]. Nano material technology has not been thoroughly explored about aspects of LCA (Life Cycle Assessment), thus in this study, we made nano silica originated from natural materials namely bamboo.

Bamboo is one of Graminae family which is very abundant in tropical countries such as Indonesia with 160 species [15]. There are 9,694,131 bamboo sticks produced throughout Indonesia [16]. One of the most widely cultivated by the community is betung bamboo. Betung bamboo sticks have many benefits, such as wood replacement construction materials, Oriented strand board (OSB), crafts and etc. In addition, the silica content contained in betung bamboo can also be utilized through proper processing. Krisdianto et al. [17] stated that there are 0.10-1.78% silica content in betung bamboo.

This study objectives were: 1) to analyze the effect of impregnation treatment using a mixture of MEG and nano silica made from betung bamboo sticks on the dimensional stability and density of sengon wood, and 2) to identify the characteristics of impregnated sengon wood.

2. Materials and Methods

2.1. Materials

Three five years old Sengon wood (Falcataria moluccana), by the diameter of 25-28 cm and originated from community forests in the Sukabumi region, West Java, Indonesia were used in this study. Other materials were MEG and nano silica from Betung bamboo sticks. Impregnation tubes and ultrasonicator were also used in this study. The logs then were cut into 2 x 2 x 2 cm$^3$ samples [18]. Weight Percent Gain (WPG), Anti Swelling Efficiency (ASE), Water Uptake (WU), Bulking Effect (BE) and density, with 5 replications for each treatment were measured.

2.2. Methods

2.2.1. Nano silica preparation from betung bamboo sticks

Betung bamboo sticks originated from community forest around Bogor. Chipped bamboo sticks, then dried until air-dried moisture content. The chips burned without fuel until they became charcoal [19]. Charcoal were put into a porcelain cup and burned at a temperature of 700°C for 6 hours in a furnace [20] to produce ash. The silica was produced from extracted ash by using the reflux method. Then, silica was mixed with PEG-6000 by using an ultrasonicator for 1 hour. Followed by burning the mixture at a temperature of 600°C for 3 hours in a furnace. The average size of nano silica made from betung bamboo sticks is 502.35 nm.

2.2.2. Impregnation process

The stages of the impregnation process are seen in Figure 1. This stage was adapted from [9].
2.2.3. Preparation of impregnation solution

MEG solution and nano silica were mixed using a Cole Parmer ultrasonicator device, with an amplitude of 40% for 1 hour. The composition of the mixture of MEG and nano silica solutions is shown in Table 1.

| Treatment               | MEG (mL) | Water (mL) | Nano Silica (g) |
|-------------------------|----------|------------|-----------------|
| Untreated (water treated)| 0        | 1000       | 0               |
| MEG                     | 500      | 500        | 0               |
| MEGsilica 0.5%          | 500      | 495        | 5               |
| MEGsilica 0.75%         | 500      | 492.5      | 7.5             |
| MEGsilica 1%            | 500      | 490        | 10              |

2.2.4. Dimensional stability and density

Dimensional stability is measured using WPG, ASE, WU and BE parameters. The weight percent gain (WPG) measurement formula is as follows:

\[
\text{WPG} (%) = \left( \frac{W_1 - W_0}{W_0} \right) \times 100
\]

where, \( W_0 \) = Oven dried weight of the sample before impregnation (g) \( W_1 \) = Oven dried weight of the sample after impregnation (g)

Anti-swelling efficiency (ASE) measurements were carried out using a 24 hours water immersion method with 2 cycles. The ASE formula is as follows:

\[
\text{ASE} (%) = \left( \frac{\text{Su} - \text{St}}{\text{Su}} \right) \times 100
\]

where, \( \text{Su} \) = Volume shrinkage of the untreated sample (%), \( \text{St} \) = Volume shrinkage of the treated sample (%)

Figure 1. The impregnation process using MEG and nano silica solutions
Measurement of water uptake (WU) after soaking in water for 24 hours is calculated by the formula:

\[
WU(\%) = \frac{(W_2 - W_1)}{W_1} \times 100
\]

where, \( W_1 \): Oven-dried weight of the sample after impregnation (g) \( W_2 \): Weight of the sample after soaking in water (g)

Bulking effect (BE) measurement formula:

\[
BE(\%) = \frac{(V_1 - V_0)}{V_0} \times 100
\]

where, \( V_0 \): Oven-dried volume of the sample before impregnation (cm\(^3\)) \( V_1 \): Oven-dried volume of the sample after impregnation (cm\(^3\))

Measurement of density (\( \rho \)) is calculated at the time before and after the impregnation treatment, with the formula:

\[
\rho = \frac{W_{\text{sample}}}{V_{\text{sample}}}
\]

where, \( W_{\text{sample}} \): Weight before and after impregnation treatment (g), \( V_{\text{sample}} \): Volume before and after impregnation treatment (cm\(^3\))

2.2.4.1. Characterization of sengon wood after impregnation

The characterization of the impregnated Sengon wood was carried out with SEM (Scanning Electron Micrograph), SEM-EDX (Scanning Electron Micrograph-energy X-ray spectroscopy), XRD (X-ray diffraction), and FT-IR (Fourier Transform Infrared Spectrometry) tools.

2.2.5. Data analysis

The experimental design used was a simple randomized complete design (CRD) which was then analyzed by ANOVA. The test was carried out using the IBM SPSS Statistics (Statistical Package for service solutions) version 25.0 calculation program, and continued with the Duncan test at \( \alpha = 1\% \) if there were real differences, the equation model used [21] was as follows:

\[
Y_{ij} = \mu + \tau_i + \epsilon_{ij}
\]

where:

- \( i = 1, 2, \ldots, t \) and \( j = 1, 2, \ldots, r \)
- \( Y_{ij} \): response or observation value from the concentration treatment of the i-solution and the j-replication
- \( \mu \): general average
- \( \tau_i \): the effect of the i-concentration treatment
- \( \epsilon_{ij} \): the effect of experimental errors from the concentration treatment of the i-impregnation solution and the j-replication
3. Results and Discussion

3.1. Dimensional stability and density

3.1.1. Weight Percent Gain (WPG)
WPG is a percentage ratio of the weight of impregnated wood to its initial weight. WPG results were shown in Figure 2. Figure 2 shows that the highest level of WPG is the MEG silica 1% treatment (73.55%). The WPG values tend to increase as the increase concentration of nano silica in the impregnation solution. It was due to the pressure in impregnation process caused MEG and nano silica entered the cell cavities and the sengon wood pits. This is in accordance with [5] which states that using a polymer at the time of impregnation causes the penetration of the polymer into the cell wall, because the polymer is able to fill pits in the cell wall. The results of the analysis of variance (p<0.05) in Table 2 indicates that treatment had a significant effect on WPG. Duncan's further test (Figure 2) shows that the concentration of each treatment was significantly different from one another.

![Figure 2](image_url)

**Figure 2.** Weight percent gain values of various MEG and nano silica impregnation treatments on sengon wood

| Source | WPG | ASE | WU | BE | Density |
|--------|-----|-----|----|----|---------|
|        | F-value | F-value | F-value | F-value | F-value |
| Treatment | 1104.74** | 54.26** | 56.82** | 180.38** | 23.47** |

S*p ≤ 0.05 indicates that treatment factor had a significant effect at a 95% confidence level

3.1.2. Anti-Swelling Efficiency (ASE)
The dimensional stability of wood is the ability of wood not to swell and shrink although there were fluctuating temperature and humidity in the surroundings [22]. Dimensional stability can be measured by anti-swelling efficiency (ASE). ASE test results are shown in Figure 3.

![Figure 3](image_url)

**Figure 3.** Anti-Swelling Efficiency values of various MEG and nano silica impregnation treatments on sengon wood
Figure 3 shows that ASE values increase as the concentration of MEG silica solution increase. The lowest ASE value is untreated (water treated) samples (8.34%) and the highest in MEG silica 1% treatment at 58.13%. The results of the analysis of variance (p<0.05) in Table 2 indicates that the ASE value had a significant influence on the concentration level of the treatment. Duncan's further test (Figure 3) states that almost every treatment concentration is significantly different from the others. ASE values are directly proportional to WPG values. The polymer enters the cell wall and covers the empty cavities that are normally occupied by water, so that the polymer coverage in wood could enhance wood dimensional stability [5]. Also, we observed that the higher WPG values were followed by the increase of ASE values. The results of this study are in accordance with [23] who states that the higher the WPG value on rubberwood, the higher the ASE value produced.

3.1.3 Water Uptake (WU)
Water Uptake is a percentage of the ability of wood to absorb water. The lower the WU value, the lower the water uptake in wood. WU results are presented in Figure 4.

Figure 4 shows the highest water uptake value is untreated (water treated) (120.36%), while the lowest is MEGsilica 1% (48.69%). The higher nano silica concentration, the lower WU values. The analysis of variance (p <0.05) in Table 2 shows that the WU value was significantly affected by the concentration level of the treatment. Duncan's further test results (Figure 4) state that almost all treatments are significantly different from other treatments. The WU results in this study are in accordance with the statement of [9] that there is a negative correlation between the value of WPG and WU, an increase in WPG is accompanied by a decrease in WU because the number of MEG and MEGsilica that cover pits in the cell wall, therefor reduces the availability of space for water molecules.

3.1.4 Bulking Effect (BE)
Bulking effect is a percentage ratio of oven-dried wood volume before and after impregnation treatment. The results of BE testing as shown in Figure 5. Figure 5 indicates that the lowest BE value was found in untreated (water treated) sengon wood (1.38%), while the highest was MEG silica 1% (12.90%). The higher nano silica in wood samples resulted the higher BE value. It was due to the coverage of MEG and nano silica on cell cavities and pits. The results of the analysis of variance (p<0.05) in Table 2 shows that the sengon wood with the concentration level of treatment significantly affected the value of BE produced. Duncan's further test (Figure 5) shows that almost every treatment concentration gives a significant different to other treatments. The results of BE in this study are in line with [5] who states that dimensional stability is increased due to polymer materials penetrate into cell walls through the diffusion process and also supports by the statement of [24] who states that wood modification using silica material is able to penetrate the cell walls so as to achieve a certain bulking effect.
3.1.5 Density
Density is a ratio of weight to volume of wood [25]. The density value obtained is presented in Figure 6.

Figure 6. Density values for various MEG and nano silica impregnation treatments on sengon wood.

Figure 6 shows that the density of sengon wood tends to increase with increasing MEGsilica concentration. The lowest density value was untreated (water treated) sengon wood (0.29 g/cm$^3$), while the highest was MEGsilica 1% (0.43 g/cm$^3$). Based on the results of the analysis of variance (p <0.5) in Table 2 shows the treatment of sengon wood significantly affected the density value. Duncan's further test (Figure 6) states that almost every treatment is significantly different from one another. The resulting density value increases along with the increase in the concentration of nano silica. It is assumed that MEG and nano silica are able to fill cell cavities and pits in the cell walls of sengon wood. This is consistent with what was stated by [26] that the higher the density of wood, the more wood content in the cell wall which means the thicker the cell wall. The density value is directly proportional to the value of WPG and ASE produced, the higher the value of WPG and ASE, the higher the density value in sengon wood.

3.2. Characterization of sengon wood from impregnation results
3.2.1. Scanning Electron Microscopy analysis (SEM)
SEM analysis (Figure 7a and 7b) shows the morphology of untreated sengon wood with magnification of 100x and 550x. We could observed that many empty pits on cell walls.
Figure 7 Scanning Electron Micrograph of untreated sengon wood vessel wall with magnification of (a) 100x and (b) 500x

Most of the pits in the vessel wall was seen to be filled with MEG, however there were some empty pits were observed due to the uneven distribution of MEG in the sample (Figure 8a and 8b). The 550X magnification in Figure 8 shows that there are several pits that were still not covered by the MEG (Figure 8b - arrowhead). Differences in the characteristics of untreated and MEG treated sengon wood can be clearly seen. The average pit size after being treated by MEG (309.80 μm) were smaller than untreated sengon wood (373.37 μm) [9]. It was due to there were some MEG covered the pits.

Figure 8. Scanning Electron Micrograph of MEG treated sengon wood vessel wall with magnification of (a) 100x and (b) 500x
Figure 9a and 9b show MEG silica 0.5% treated samples with 100x and 500x magnification, respectively. Nano silica and MEG can cover and fill pits on cell walls. However, we could observe that there were some pits still not covered by nano silica due to small concentration of nano silica used during the impregnation process (Figure 9b-arrow).

Figure 9. Scanning Electron Micrograph of MEGsilica 0.5% treated sengon wood vessels wall with magnification of (a) 100x and (b) 500x

Figure 10. Scanning Electron Micrograph of MEGsilica 0.75% treated sengon wood vessel wall with magnification of (a) 100x and (b) 500x
Figures 10 and 11 show MEG and nano silica 0.75% and 1%, respectively. MEG and nano silica can enter and fill the lumen cells and pits of the sengon wood vessel. There are nano silica agglomeration caused by more concentration of nano silica in these treatments. Agglomeration in MEGsilica 0.75% (Figure 10b - arrows) were less than MEGsilica 1% (Figure 11b - arrows).

![Figure 11. Scanning Electron Micrograph of MEGsilica 1% treated sengon wood vessel wall with magnification of (a) 100x and (b) 500x](image)

The SEM analysis results show the reasons why improvement values of WPG, BE, ASE, density and WU occurred in treated sengon wood. According to Xue and Zhao [27] nano particles can enter and penetrate into wood mainly through nano-sized cavities.

### 3.2.2 SEM-EDX Analysis

The SEM-EDX analysis results in Table 3 shows that there was no silica content in untreated (water treated) and MEG treated. In contrast to wood samples impregnated with MEGsilica 0.5%, MEGsilica 0.75% and MEG silica 1%. The silica content increases with increasing nano silica concentrations. It can be concluded that nano silica deposits in wood cells.

| Treatment (wt.%)                  | Silica (wt.%) |
|----------------------------------|---------------|
| Untreated (water treated)        | 0             |
| MEG                              | 0             |
| MEGsilica 0.5%                   | 1.93          |
| MEGsilica 0.75%                  | 3.85          |
| MEGsilica 1%                     | 5.52          |

**Table 3.** Chemical composition of sengon wood samples using SEM-EDX analysis on various MEG and silica impregnation treatments
3.2.3 X-Ray Diffraction (XRD) analysis
XRD testing is carried out at an angle of $2\Theta$ between 5°-40°. Based on the results of the XRD analysis (Figure 12), there is a peak with an angle of $2\Theta = 21.81^\circ$-22.87°. According to the JCPDS data, the peak is plane 002 indicating the crystalline phase of cellulose. Then there is a peak with an angle of $2\Theta = 16.97^\circ$ which is the peak of cellulose I$\beta$ in the diffraction plane 1101. In accordance with [28] states that cellulose I$\beta$ appears at an angle of $2\Theta = 14$-16°.

![Figure 12. Sengon wood XRD spectra of various treatments](image)

**Figure 12.** Sengon wood XRD spectra of various treatments

Additional information about XRD testing can be seen in Table 4. There was a decrease in the degree of crystallinity in the sengon wood sample impregnated by MEG and nano silica. This is caused by MEG which is amorphous and nano silica which is suspected semi-crystalline because of the sonication treatment that were used. This was in line with Dirna [29] who performed impregnation by using MEG and nano silica from betung bamboo leaves. This result is also supported by Dong et al. [30] which states that the crystallinity of poplar wood decreases after the addition of amorphous furfuryl alcohol. XRD analysis shows the effect of nano silica to WPG, ASE, WU, BE and density.

| Treatment                  | Degree of crystallinity (%) |
|----------------------------|------------------------------|
| Untreated (water treated)  | 23.67                        |
| MEG                       | 24.00                        |
| MEGsilica 0.5%             | 23.31                        |
| MEGsilica 0.75% F         | 17.98                        |
| MEGsilica 1%              | 21.39                        |

3.2.4. Fourier Transform Infrared Spectrometry (FT-IR) analysis
Fourier Transform Infrared Spectrometry (FT-IR) is an infrared spectroscopy technique that can analyze structural polysaccharides and observe structural changes in biopolymers [31]. Based on Figure 13, it can be seen that the absorption peaks of the infrared spectrum have a significant difference between untreated and treated sengon wood. Absorption bands ranging from 3354.31 to 3375.43 cm$^{-1}$ are hydroxyl groups (O-H) from water in sengon wood. In accordance with the statement of Dachriyanus [32] that in the wave frequency range between 3750 and 3000 cm$^{-1}$ it was detected that there was an O-H group. However, in different sengon wood treated with MEG silica there was no peak at wave 478.30 cm$^{-1}$ which is the bending of the Si-O-Si group, this is because silica does not bond to the cell wall components of the sengon wood. At wave 1057 cm$^{-1}$, C-O groups appearing from MEG which appear new functional groups such as furan rings at waves 1027.67 cm$^{-1}$ and 778.62 cm$^{-1}$ [32,33]. The C = O
function group of the sengon wood hemicellulose appeared at wave 1737.32 cm$^{-1}$. Pangau et al. [33] state that the C = O group appears in waves ranging from 1723.48 to 1742.53 cm$^{-1}$.

Figure 13. FT-IR spectrum of various sengon wood treatments

MEG and nano silica do not bond with the cell wall components of sengon wood which could cause MEG silica to be leached by water. MEG silica is only able to fill the cell wall cavities which gives a bulking effect on the MEG silica treated sengon wood.

4. Conclusion
The MEG and nano silica of betung bamboo sticks impregnation treatment has a significant effect on the dimensional stability and density of the sengon wood. Increasing values of WPG, ASE, BE, and density as well as decreasing the value of WU shows the polymer can cover the lumen cell and pits of sengon wood. The morphological changes of sengon wood after MEG and nano silica impregnation treatment (based on SEM images) showed that the lumen cell and pits were occupied by MEG and nano silica. MEG and nano silica are bulking agents. Based on research, the optimum treatment for five years old sengon wood was MEG silica 1%.

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