

Communication

# Timber Properties and Cellulose Crystallites Size in Pine Wood Cut in Different Sawing Patterns after Pretreatment with $\text{CH}_3\text{COOH}$ and $\text{H}_2\text{O}_2$ and Densification

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**Abstract:** One process to improve wood quality is densification or wood surface compression. Our study analyzed the changes in some basic properties of pine wood, including its anatomical structure, density, modulus of elasticity (MOE), and dimensions of cellulose crystallites, after densification following soaking pretreatment in  $\text{CH}_3\text{COOH}$  and  $\text{H}_2\text{O}_2$  at a concentration of 20%. Samples were sawn in radial and tangential directions for analysis of the wood. The results showed a change in the shape of tracheid cells from hexagonal to oval, as well as damage to the ray cell constituents on the tangential surface. The thickness decrease of the samples was in accordance with the target, which meant that spring-back was short. In general, the tangential boards had a higher density than the radial boards, with a lower MOE and crystallite dimensions. Our findings showed that the densified tangential board was stronger than the radial board.

**Keywords:** densification; soaking;  $\text{CH}_3\text{COOH}$ ;  $\text{H}_2\text{O}_2$ ; radial; tangential



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## 1. Introduction

The availability of wood from natural forests is becoming limited, which has affected forest product industries and led to increased use of wood from plantation forests. However, wood from plantation forests is known to have lower quality than wood from natural forests because it is generally harvested at a young age. Consequently, it has a high portion of juvenile wood and thus low durability.

According to Panshin and de Zeeuw [1] and Bowyer et al. [2], juvenile wood is characterized by low specific gravity and density, short fibers, a large amount of longitudinal shrinkage, and a mostly flatter microfibril angle. It also has less dimensional stability and lower amounts of the chemical components that compose cellulose, but it has a higher lignin content than mature wood. Owing to these characteristics, the processing is needed to improve the quality of wood from fast-growing wood species from plantation forests.

One of the processes for improving the quality of wood is densification or wood surface compression. According to Neyses [3], densification is the process of compressing the wood surface, which results in increased density and hardness of the wood surface in part or all of the densified material. One of the parts of the densification process is the pre-treatment before the densification process.

Neyses et al. [4] reported that pretreatment with a solution of sodium silicate, sodium hydroxide, methacrylate resin, and an ionic liquid increased the surface hardness of pine wood. Previous research by Yunianti et al. [5] showed that pretreating wood by soaking it in a mixture of  $\text{CH}_3\text{COOH}$  and  $\text{H}_2\text{O}_2$  increased wood density. They also found that modifying temperature and time parameters within the densification process resulted in different responses in pine and gmelina wood. Pretreatments using high-temperature immersion [6] and thermal compression under several conditions [7] also influenced wood modification.

In addition to the increase in density, densification changes the anatomical structure of the wood. In particular, it alters the shape of vessels and causes tracheids to become more oval [8–10], which can affect wood properties. However, it is important to consider that wood is an anisotropic material that generally has different behaviors and responses based on the three directions of the wood grain. Grain direction refers to the longitudinal/axial direction, which is parallel to the tree trunk axis, and the radial and tangential directions, which are perpendicular to the tree trunk axis. The differences in the behaviors and responses of wood are caused by the arrangement of cells wood grain. Cell arrangement affects the anatomical structure of wood, which in turn affects the other wood properties. The anisotropy of wood is particularly notable in light of the sawing patterns used to cut logs into wood boards. Previous studies on wood boards used as samples in testing did not report the sawing pattern, and this omission does not allow for determining the different responses of a wood type receiving the same pre-treatment before the densification process.

In addition, the increase of wood density in the densification process is thought to be influenced by the cell wall ultrastructure, including the microfibril angle and cellulose crystallites. According to Stuart and Evans [11], Butterfield [12], Saranpää [13], Peura et al. [14], Lachenbruch et al. [15], Yin et al. [16], and Tabet and Fauziah [17], the microfibril angle and cellulose crystallites are the main factors affecting the physical properties of wood (especially density and shrinkage), its mechanical properties (especially tensile strength and stiffness), and its chemical content.

Knowledge is needed on the changes in the characteristics of cellulose crystallites after the densification process. Such knowledge could help determine the success of the densification process. It could also explain different responses, especially in wood that has the same density or the same cell wall thickness. In addition, the sawing pattern, along with different cell types, will affect how wood responds to treatment before and during the densification process. Therefore, this study assessed the changes in some basic properties of wood, including anatomical structure, density, modulus of elasticity (MOE), and dimensions of cellulose crystallites, after the densification process. Wood was cut with different sawing patterns and underwent a pre-soaking treatment in a solution of  $\text{CH}_3\text{COOH}$  and  $\text{H}_2\text{O}_2$ , following previous research by Yuniarti et al. [5].

## 2. Materials and Methods

### 2.1. Wood Materials

This study used pinewood (*Pinus merkusii*) that was cut into 1-m-long logs, which were subsequently cut into tangential and radial boards. The size of each board was 100 cm (L)  $\times$  25 cm (W)  $\times$  2 cm (H). Boards were then cut into defect-free samples that were 30 cm (L)  $\times$  25 cm (W)  $\times$  2 cm (H) in size. Ten samples were prepared for each tangential and radial board, of which five samples were densified and did not treat the others.

### 2.2. Densification Process

Densification was done following the process reported by Yuniarti et al. [5]. The wood samples were pre-treated by soaking in a solution of  $\text{CH}_3\text{COOH}$  and  $\text{H}_2\text{O}_2$  at a concentration of 20%. Afterward, the pre-treated samples were densified under a pressure of 35 kg/cm<sup>2</sup> at 150 °C for 30 min to obtain a thickness reduction of about 30%. All the densified wood samples were conditioned for 24 h in a desiccator and stored until use in the following experiments. Untreated wood samples were also prepared for use as a control group.

### 2.3. Sample Preparation

The untreated and densified treated wood samples were cut into various sizes depending on the experiments in which they were to be used. For microscopic observation and measurement of density and set recovery, samples were cut to 2 cm  $\times$  2 cm  $\times$  1.4 cm. The samples for MOE testing were 2 cm  $\times$  1.4 cm  $\times$  30 cm. In addition, for the measurement of cellulose crystallites dimensions, samples were ground to pass 40 mesh.

#### 2.4. Microscopic Observation

Microscopic observation was undertaken to determine changes in cell shape following the same preparations method as in the analysis of wood anatomy. The observations were made with regard to the three directions of the wood grain (axial, tangential, and radial) from the radial and tangential boards.

#### 2.5. Recovery of Set

The measurement of set recovery was carried out on densified wood samples with a thickness reduction target of 30%. The set recovery (SR) is defined as:

$$\text{SR (\%)} = \frac{R'_C - R_C}{R_0 - R_C}$$

where

$R_0$  = Oven-dry dimension of the specimen in the densification direction before densification (mm),

$R_C$  = Dimension of the specimen after densification (mm),

$R'_C$  = Dimension of the densified sample after the wet-dry cycling (mm).

#### 2.6. Wood Density Measurement

The density was measured under air-dry conditions according to British Standard Methods No. 373 [18] with the modification of sample size due to the changes in thickness that occurred after the densification process. The measurement of wood used the dip method. The density ( $\rho$ ) was calculated as follows:

$$\rho = \frac{m}{v}$$

where

$m$  = weight of sample under air-dry conditions (g),

$v$  = volume of sample under air-dry conditions (cm<sup>3</sup>).

#### 2.7. Modulus of Elasticity Measurement

The MOE was determined under air-dry conditions according to ASTM D 143-94 [19] with a modification of sample size due to changes in thickness that occurred after the densification process. The equation for calculating MOE was as follows:

$$\text{MOE} = \frac{\Delta PL^3}{4\Delta\delta bd^3}$$

where

$\Delta P$  = loading increment in the proportional section of the load deflection diagram (elastic zone) (kg);

$L$  = span (cm);

$\Delta\delta$  = deflection measured at mid-span (cm);  $b$  = width of sample (cm);  $d$  = thickness of sample (cm).

#### 2.8. Measurement of Dimensional Crystallite

The measurement of dimensional crystallites used X-ray, with CuK ( $\lambda = 0.154$  nm) as the radiation source at 40 kV and 30 mA with a diffraction angle ( $2\theta$ ) and measured between  $20^\circ$  until  $40^\circ$  with a speed at  $2^\circ/\text{min}$ . The crystallite width was measured at reflection 200

( $2\theta = 24.4^\circ$ ) and the length was measured at reflection 004 ( $2\theta = 34.4^\circ$ ). The average width and length of crystallites ( $D_{hkl}$ ) were calculated according to the Scherrer formula [14]:

$$D_{hkl} = \frac{K\lambda}{\beta \cos \theta_{hkl}}$$

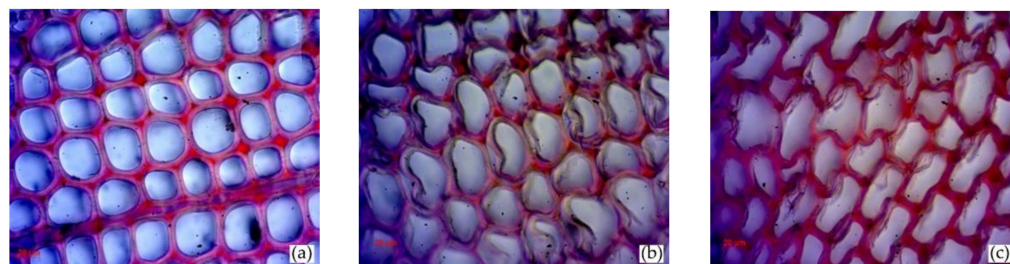
where  $D_{hkl}$  = dimensions of the crystallites (nm);  $\lambda$  = wavelength targets (copper = 0.154 nm);  $\beta$  = FWHM in radians;  $\theta$  = half of the scattering angle  $2\theta_{hkl}$ ;  $K$  = Constant for graphite sheets (0.9 for reflection 200 and 1.0 reflection 004).

### 3. Results and Discussion

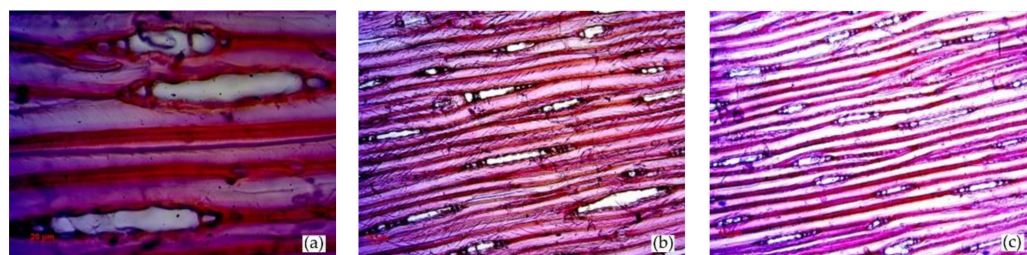
Results of this study indicated changes in the anatomical structure, thickness (set of cover), density, and MOE of radial and tangential boards as an effect of the densification treatment. Different sawing patterns were associated with different changes in several characteristics of the basic properties of pinewood.

#### 3.1. Changes in Anatomical Structure

According to Frans [20], a solution of  $H_2O_2$  and  $CH_3COOH$  at a certain concentration can soften the wood and separate the individual cells of wood fibers. In our study, samples soaked in a solution of  $H_2O_2$  and  $CH_3COOH$  at a concentration of 20% for 24 h at  $80^\circ C$  prior to a densification process that used a pressure of  $35 \text{ kg/cm}^2$  at a temperature of  $150^\circ C$  for 30 min, causing changes in the pine wood constituent cells. The hexagonal tracheids became oval shaped on the axial, radial, and tangential surfaces (Figure 1), and the ray cells were damaged on the tangential surface (Figure 2). According to Brauns and Rocens [21] and Yunianti and Suhasman [10], during the densification process tracheid/vessel cells become oval shaped because of the compression of the cell wall.



**Figure 1.** The changes of tracheid cell, (a) control, (b) radial surface, and (c) tangential surface, under high magnification ( $\times 40$ ).



**Figure 2.** The damage of the ray cells on the tangential surface of the radial and tangential boards (a) under high magnification ( $\times 40$ ), and (b,c) under low magnification ( $\times 10$ ).

In the tangential board, the pressure was applied in the radial direction, where the transverse ray cells are present. The damage mostly affected the tracheid cells, which have a larger diameter than the ray cells. Owing to the larger diameter, the tracheids have more space and therefore become denser when pressed. In the radial boards, the pressure was applied in the tangential direction. In general, the damage or flattening was related to the

ray cells, which have a small diameter. Therefore, less free space was lost when the board was pressed.

When the pressure occurs in the radial direction, the strength is in the ray cell, and when the pressure occurs in the tangential direction, the strength is in the sapwood [22]. Consequently, wood in the radial and tangential boards responded differently during the densification process. In addition, the damage to cells and the changes to wood properties were also different. According to Nairn [23], during the pressing process, the response of the wood depends on the density, percentage of sapwood, the volume of the ray cells, and the direction of the pressure surface. In the current study, the first damage occurred in the sapwood and continued to the whole layer.

### 3.2. Thickness Changes (Set of Recovery)

According to Neyses et al. [4], set of recovery is a dimensional change in the thickness of densified wood, measured from the initial dimensions and the dimensions after removal of the deformation. The set of recovery can be reduced by chemical modification. In our study, pre-treatment was based on soaking the wood samples in a 20% solution of  $H_2O_2$  and  $CH_3COOH$  before the densification process softened the wood due to the separation of individual cells. Based on previous research by Yunianti et al. [5], the 20% concentration was associated with the best quality of densified wood compared with the other concentrations.

In our study, pre-treatment based on soaking the wood samples in a 20% solution of  $H_2O_2$  and  $CH_3COOH$  before the densification process softened the wood due to the separation of individual cells. Based on previous research Yunianti et al. [5], the 20% concentration was associated with the best quality of densified wood compared with the other concentrations.

The thickness changes of the radial boards and the tangential boards of pinewood after the densification process were in accordance with the target of thickness reduction of 30%. The decreased thickness of the tangential board was 32%, whereas the radial board thickness decreased by 31%. This outcome indicates that the densified pinewood did not have much spring back and tended to be stable. According to Rautkari [24], Hartono et al. [25], and Báder and Németh [26], some wood component cells sustain shape changes during the densification process. If the cells return to their original shape when the pressure is removed, spring back has occurred.

### 3.3. Density

Figure 3 shows that wood density increased from 11.76% to 18.91% in the radial and tangential boards, respectively. This research result is in line with a previous report by Mania et al. [27], who found that the density of poplar and birch wood increased after the densification process.

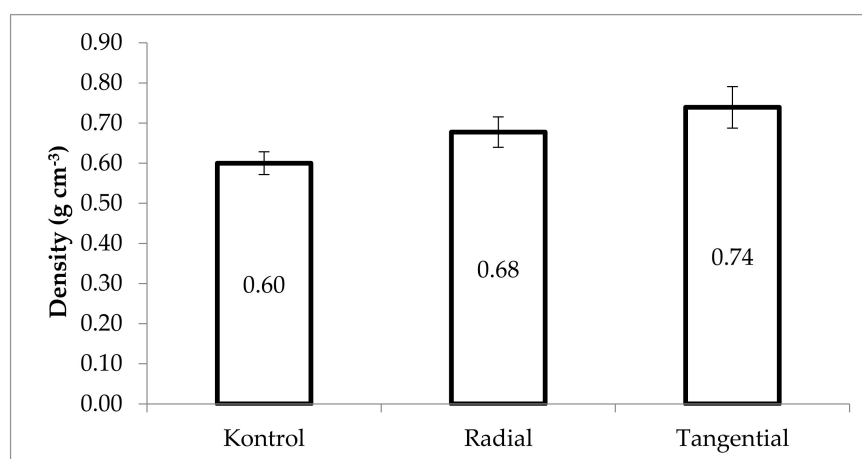


Figure 3. The Density of Pine Wood from Radial Board and Tangential Board.

According to Rautkari [24], a shorter time in the pressing process along with a lower temperature will cause the density on the wood surface to increase. In the current study, the densification process at a temperature of 150 °C for 30 min generated a change in the density of radial and tangential boards.

The increased density was associated with the different sawing patterns, which were closely related to the changes in the densified wood cells. The increased number of cells per unit of the same area indicated the cause of the density change in the densified wood. The damage to the tracheid cells caused the increased number of cells per unit of the same area, and therefore, the tangential board was found to have a higher density than the radial board. Meanwhile, since the ray cells became the most damaged cell on the radial board the area with these cells was smaller than in the tangential board. The increase in density is influenced by the anisotropic properties of wood [22].

### 3.4. Changes in MOE

Figure 4 shows that MOE of the radial and the tangential boards increased after pinewood was pretreated with 20% H<sub>2</sub>O<sub>2</sub> and CH<sub>3</sub>COOH and densified.

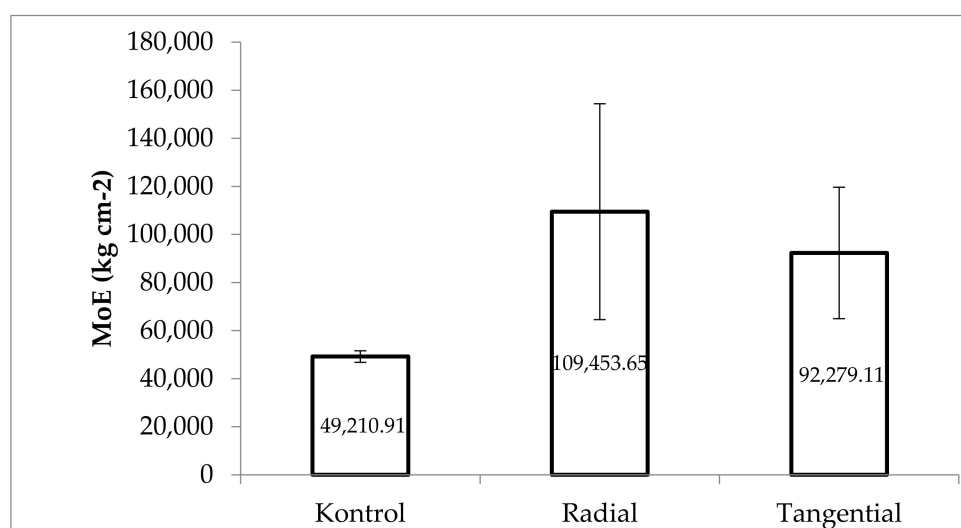


Figure 4. MOE of densified Pinewood on The Radial Board and The Tangential Board.

According to Brauns and Rocens [21], the different relations between wood elasticity and densified wood occurs in longitudinal, radial, and tangential directions. The strength increases as the wood elasticity increases. The relation varies depending on the type of wood and the method of densification used. In our study, the MOE value increased in the radial and tangential boards, which was in line with the increase in the wood density. According to Blomberg [28], semi-isostatic densified wood resulted in low cracking rates and high strength, even in an irregular shape. The deformation is determined by the anisotropic properties of wood.

### 3.5. Changes in Dimensions of Crystallites

Cellulose crystallites affect the physical, mechanical, and chemical properties of lignocellulosic materials. The crystalline part of the wood is the fraction of the densely crystalline material of cellulose microfibrils, and it is affected by the length and width of the crystallites [29]. The crystallite dimensions have been reported for some conifers by Andersson et al. [29], Anderson [30], and Peura et al. [14], and their results showed that the crystallite length ranges from 6.50 to 36.40 nm, and the width ranges from 2.5 to 3.60 nm. Our study showed that the width and length of the crystallites differed between radial boards and tangential boards following pre-treatment and densification (Table 1).

**Table 1.** Changes in crystallite dimensions in radial and tangential boards after densification.

Crystallite Width (nm)		
Sawing Pattern	Before Densification	After Densification
Radial	2.22 ± 1.25	1.30 ± 0.16
Tangential		1.31 ± 0.19
Crystallite Length (nm)		
Sawing Pattern	Before Densification	After Densification
Radial	6.14 ± 3.88	3.78 ± 0.26
Tangential		3.26 ± 0.18

Changes in the dimensions of the crystallites (i.e., length and width) can especially indicate changes in the density of the wood. The smaller the cellulose crystallite dimensions, the higher the density. These changes may be related to the chemical components of cell walls, especially cellulose. According to Panshin and de Zeeuw [1], crystallites are part of the crystalline microfibrils formed from cellulose chains. In addition, according to Kutnar and Sernek [31], chemical densification causes changes in the chemical components of cell walls and is formed as covalent bonds. The soaking treatment of H<sub>2</sub>O<sub>2</sub> and CH<sub>3</sub>COOH caused the composition of the chemical components in cells to change, which led to changes in the dimensions of the crystallites in the cell walls. In this research, the change in the dimensions of the cellulose crystallites was associated with the increased density of the radial and tangential boards.

#### 4. Conclusions

In association with pre-treatment using a solution of CH<sub>3</sub>COOH and H<sub>2</sub>O<sub>2</sub> at a concentration of 20% before the densification process, the radial and tangential boards showed changes in the shape of tracheid cells from hexagonal to oval and damage to the ray cell constituents on the tangential surface. The thickness decreased in accordance with the target amount (30%), indicating the occurrence of short spring back. In general, tangential boards have a higher density (0.74 g/cm<sup>3</sup>) than radial boards (0.68 g/cm<sup>3</sup>), with a lower MOE and smaller crystallite dimensions. Therefore, a tangential board is stronger than a radial board after the densification process.

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