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Radial Variations in Surface Characteristics of Pine Wood (*Pinus merkusii*) Modified with Glycerin–Citric Acid

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ABSTRACT

Pine wood (*Pinus merkusii*) is a native Indonesian softwood species with high commercial value but low natural durability (class IV), limiting its use in outdoor applications. This study modified the pine wood using a 20% glycerin–citric acid solution (1:2 ratio) through impregnation with 7 bar pressure for 48 hours, followed by heat treatment at 150°C for 6 hours, to evaluate surface characteristics in radial variations (from pith to bark). Retention (57.93–85.59 kg/m³) and weight percent gain (WPG, 4.02–7.20%) decreased from pith to bark, corresponding to lower wood density near the pith (0.55 g/cm³) compared to near the bark (0.67 g/cm³). Surface roughness (Ra) varied slightly from fit to bark, however its Ra increased after modification. Surface free energy (SFE) increased significantly from 16.87 near the pith to – 27.35 mJ/m² near the bark. After modification the SFE values varied between 37.16–44.46 mJ/m² from fit to bark. Wettability (K-value) was lower near the pith (0.002) than near the bark (0.007). Glycerin–citric acid modified pine exhibited also a lower wettability values compared to the untreated wood. The surface characteristics of the modified pine wood from fit to bark indicate that its surfaces should be coated by proper varnishes or paints for further development.

Keywords: citric acid, glycerin, modification, radial variation, surface characteristics

INTRODUCTION

Pinus merkusii is a type of softwood belonging to the Pinaceae family and is the only pine species native to Indonesia. Pine trees have high economic potential, both in terms of wood and non-wood products. Pine wood can be utilized in various sectors, such as for light construction, furniture manufacturing, and pulp raw material. Additionally, non-wood products such as sap or resin are processed into derivative products like rosin and turpentine, which have high market value (Suluh and Sampelawang 2017). The production of pine logs increased from 145,896 m³ in 2018 to 226,391 m³ in 2022 (BPS 2023). Pine wood has a specific gravity of 0.40–0.75 (average 0.55) and belongs to strength class III. However, it has a low natural durability, classified as durability class IV (Seng 1964). This low durability makes pine wood susceptible to damage by wood-degrading organisms, has low dimensional stability, and can be degraded by sunlight through photodegradation. Due to these natural characteristics, pine wood is less resistant to biological and environmental degradation, hence requiring modification treatments to improve its service life. Such treatments may include the application of preservatives into the wood and surface finishing (Lestari 2020).

Wood quality enhancement can be achieved through impregnation with chemical preservatives. The selection of wood modification methods today is increasingly based on their environmental impact. As global concern for environmental issues continues to rise, the use of chemically based modification techniques has begun to decline (Lestari *et al*. 2023). Chromated Copper Arsenate (CCA) was previously widely used as a wood preservative due to its effectiveness against insects and microbes, but it has adverse environmental impacts (Meena 2022). The arsenic content in CCA is toxic to aquatic and soil organisms and is carcinogenic, posing risks to body systems upon exposure (Morais *et al.* 2021). Therefore, CCA has been gradually phased out and replaced by more environmentally friendly substances, such as glycerin and citric acid. Lopes *et al.* (2025) stated that citric acid effectively inhibites termite feeding behaviour, it repels termites or creates conditions unsuitable for their survival and feeding activities, thereby preserving the integrity of the wood. Citric acid impregnation also reduces the wood's moisture content, which can decrease the availability of water required by fungi to grow and cause decay. The lower moisture content in citric acid-impregnated wood can slow down the rate of decay, resulting in less

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damage to the wood. Meanwhile, the study by Essoua *et al*. (2016) showed that wood preservation using a combination of glycerol and citric acid is able to provide high resistance against fungal attack, with a possible release of sorption water from the wood or polymer structure during the testing process.

Environmentally friendly wood modifications have been widely studied. The modifications not only affect the physical and mechanical properties of wood but also influence its surface characteristics. Martha (2019) showed that a combination of chemical and thermal modification on short-rotation teak wood increased its dimensional stability but decreased the modulus of elasticity (MOE) and modulus of rupture (MOR). On the other hand, it also reduced surface roughness, surface free energy (SFE), and wettability as the treatment temperature increased, indicating that the modification made the surface more hydrophobic. The study by Hanifah *et al.* (2023) also demonstrated that chemical and thermal modification affected the surface roughness of short-rotation teak wood. This modification enhanced hydrophobicity, reduced surface free energy (SFE), and produced K values approaching zero, indicating that water-based paints have difficulty spreading and penetrating the surface of the treated wood.

The radial cross-section of a stem consists of three zones: juvenile wood (JW), transition wood, and mature wood, although most studies only distinguish between juvenile and mature zones (Alteyrac *et al.* 2006). These zones exhibit distinct characteristics, as pith-to-cambium variations in anatomical traits such as ring width, latewood proportion, cell wall thickness, microfibril angle, and fibre lengths are commonly used to define juvenile and mature wood regions and estimate the transition age. These variations also correspond to differences in the physical properties of the wood (Fos *et al.* 2023).

Research specifically investigating the effects of glycerin–citric acid modification on the surface characteristics of pine wood such as surface roughness, surface free energy, equilibrium contact angle, and wettability while considering its radial variation has not previously been conducted. These aspects are important to optimize the modification process and to consider the most appropriate finishing techniques and materials to apply to the treated pine wood surface. Therefore, this study aims to determine the surface characteristics of radial variations in pine wood treated with glycerin-citric acid.

METHODS

Tools and Materials

The equipment used in this study included an impregnation chamber, compressor, digital scale, oven, desiccator, caliper, pencil, laptop, syringe, aluminum foil, surface roughness tester (©Mitutoyo type SJ-210), beakers, measuring cylinders, camera, macro capture microscope, ImageJ, GOM Player, and SAS software. The primary material used was pine wood (*Pinus merkusii*) over 50 years old, obtained from the Gunung Walat Educational Forest, Sukabumi. Additional materials used included distilled water, citric acid monohydrate (BP/USP/FCC/E330 grade, CAS No. 5949-29-1) obtained from Shandong Ensign Industry Co., Ltd., China, Glycerin (refined grade, batch no. B085-B, PT. Ecogreen Oleochemicals, Medan, Indonesia), toluene, and 50% methanol.

Sample Preparation

The pine wood used in this study was in the form of discs with a diameter of 40 cm and a thickness of 10 cm, taken at a height of 130 cm from ground level. Two discs were obtained from two different pine trees. Each disc was cross-cut from one bark side to the other, passing through the pith, with a cut width of 8 cm and the pith positioned exactly in the center. These cuts were then split radially from the pith towards the bark, with a thickness of 2 cm, resulting in small blocks measuring 2 cm × 8 cm × 10 cm.

Test samples from the first tree disc were labeled with code A, while samples from the second disc were labeled C. Each disc produced test samples labeled A1–A7 and C1–C7, starting from the position closest to the pith to the one nearest the bark, with a percentage of heartwood of approximately 18% and sapwood of 82%. Samples were divided into two types: treatment and untreated (Figure 1).

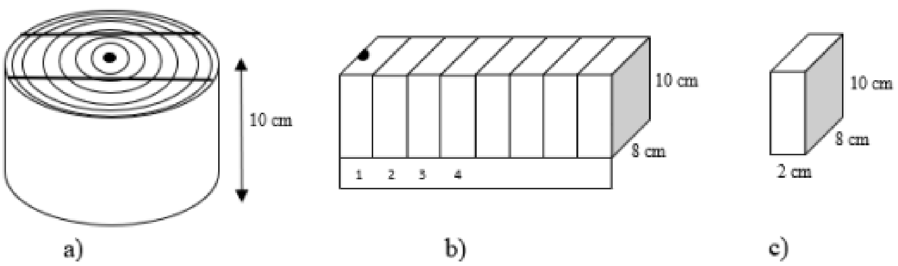


Figure 1 Sample Collection Method: a) log wood; b) lumber assortment; and c) test sample.

Modification Processes

Wood modification was carried out using a 20% solution of a glycerin–citric acid mixture through the impregnation method. The impregnation solution was prepared by mixing a 20% glycerin–citric acid solution in a 1:2 ratio with 80% distilled water, then stirred at room temperature until homogeneously mixed. The impregnation process began by applying a pressure of 7 bar for two days using the glycerin–citric acid solution. After impregnation, the samples were drained until no solution dripped. The samples were then wrapped in aluminum foil and placed in an oven for thermal modification. Thermal modification was carried out in stages: heating to 100°C for 1 hour; increasing the temperature from 100°C to 150°C; heating at 150°C for 6 hours; and cooling for 1 hour.

Retention dan WPG

Retention and WPG were calculated using the following equations:

$$\text{Retention (kg/m}^3\text{)} = \frac{B_1 - B_0}{V} \times K$$

$$\text{WPG (\%)} = \frac{W_1 - W_0}{W_0} \times 100$$

Where:
B_0 = Weight of the wood at 103 ± 2°C before impregnation
B_1 = Final weight of the wood after impregnation
V = Volume of the wood at 103 ± 2°C before impregnation
K = Concentration of the impregnation solution
W_0 = Weight of the wood at 103 ± 2°C before impregnation
W_1 = Final weight of the wood after heat treatment

Surface Roughness

The surface roughness of the samples was measured using a Mitutoyo tester, type SJ-210. The sensor was placed at five different points on the wood surface, with the measurement direction perpendicular to the grain. The measurements followed ISO 4287:1997 standards, with parameters including a cut-off length of 0.8 mm, a measurement path length of 6 mm, and a measurement speed of 0.5 mm/second. The surface roughness value used was the arithmetical mean roughness (Ra), and it was classified according to Table 1.

Determination of Equilibrium Contact Angle and Wettability Value

The equilibrium contact angle (θ_e) was determined using a segmented regression approach between time (t) and contact angle (θ) using the PROC NLM procedure in SAS software. The K value was calculated based on the S/G model (Shi and Gardner 2001) using XLSTAT software with the following equation.

$$\theta = \frac{\theta i . \theta e}{\theta i + (\theta e - \theta i)\exp\left[K\left(\frac{\theta e}{\theta e - \theta i}\right)t\right]}$$

Where:
θ = Contact angle at a given time (°)
θ_i = Initial contact angle (°)
θ_e = Equilibrium contact angle (°)
t = Time in seconds, and K is the rate constant of contact angle change

Determination of Surface Free Energy

The two-liquid method was treated into a multi-liquid method to determine the value of surface free energy (SFE) and its components, as proposed by Rabel (1971), using the regression line as follows.

$$(1 + Cos\ \theta_e)\frac{\gamma_l}{(\gamma_l^d)^{\frac{1}{2}}} = (\gamma_s^d)^{\frac{1}{2}} + (\gamma_s^p)^{\frac{1}{2}}\left(\frac{\gamma_l^p}{\gamma_l^d}\right)^{\frac{1}{2}}$$

Where:
θ_e = equilibrium contact angle
γ^L = total surface tension of the liquid
γ^{Ld} = dispersive component of the liquid's surface tension
γ^{sd} = dispersive component of the surface free energy (SFE)
γ^{Lp} = polar component of the liquid's surface tension
γ^{sp} = polar component of the SFE

The components of surface free energy (SFE) are determined from the measurement of the equilibrium contact angle θ_e of standard liquid droplets (γ^L) on the surface of the wood samples, which is then used to calculate the SFE value (γ^s). In linear regression:

Table 1 Surface smoothness classification (ISO 4287:1997)

Ra	Surface Condition
< 0.1 μm	Very Smooth
0.1 – 0.5 μm	Smooth
0.5 – 2 μm	Moderately Smooth
>2 μm	Rough

$$(Y = A + BX),$$

$$Y = (1 + Cos\ \theta_e)\frac{\gamma_l}{(\gamma_l^d)^{\frac{1}{2}}},\ X = \left(\frac{\gamma_l^p}{\gamma_l^d}\right)^{\frac{1}{2}},$$

Thus, the slope (B) is $(\gamma_s^p)^{1/2}$ and the intercept (A) is $(\gamma_s^d)^{1/2}$The values of X and Y in this study were calculated based on four standard liquids, as presented in Table 2. Meanwhile, the Y values were obtained from the contact angle measurements of the standard liquids in Table 2 on each wood specimen surface. The SFE value was calculated using the following equation:

$$A^2 + B^2 = \left((\gamma_s^p)^{\frac{1}{2}}\right)^2 + \left((\gamma_s^d)^{\frac{1}{2}}\right)^2.$$

Data Analysis

Data analysis was conducted both quantitatively and descriptively by observing patterns or trends in the data presented in graphical form, based on the average values of each tested wood surface characteristic.

RESULT AND DISCUSSION

Retention and WPG

The values of retention and WPG are presented in Figure 2. Retention indicates the amount of preservative (impregnant) absorbed into the wood per unit volume, while WPG (Weight Percent Gain) refers to the increase in wood weight or mass after chemical-thermal modification. For Tree A, retention and WPG values ranged from 4.02% to 6.37% and 69.18 to 84.05 kg/m³, respectively; for Tree C, they ranged from 5.27% to 7.20% and 57.93 to 85.59 kg/m³. Retention and WPG values in pine wood decreased from the pith toward the bark. This may occur because the wood near the pith has a lower density compared to the wood near the bark. A study by Abdussalam (2024) using similar samples showed that the lowest pine wood density was found near the pith at 0.55 g/cm³, while the highest density was near the bark at 0.67 g/cm³. Similar results were also reported by Darmawan *et al.* (2018), indicating that the density of pine wood increases proportionally from the pith to the bark.

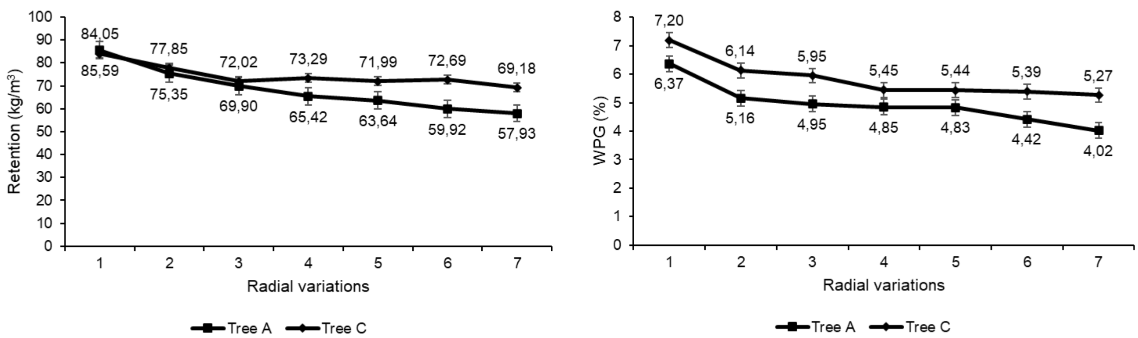
Wood located near the pith has lower density because juvenile wood is concentrated in this area (Gaol *et al.* 2023). The low density of juvenile wood is due to its thin cell walls, wide growth rings, and fewer latewood cells (Shmulsky and Jones 2019). Wood density can affect retention and WPG values. Wood with low density generally has larger and more open vessels, resulting in higher permeability compared to wood with higher density (Amin *et al.* 2021). Therefore, the retention and WPG values of wood near the pith are higher than those of wood near the bark.

Wood located near the pith has a lower density due to the concentration of juvenile wood in that area (Gaol *et al.* 2023). The low density of juvenile wood is

Table 2 Surface tension values of test liquids and their components (in mJ/m²) used in the calculation of wood surface free energy (Yuningsih *et al.* 2019)

Liquids	γ_l^p	γ_l^d	γ_l
Water	21.8	51	72.8
Methanol 50%	12.9	22.7	35.6
Toluene	2.3	26.1	28.4
Glycerin	30	34	64

Remaks: γ_l^p = Polar component of the surface tension γ_l^d = Dispersive component of the surface tension and γ_l= Total surface tension value.



(a) (b)

Figure 2 Retention (a) and WPG (b) values of untreated and treated pine wood from pith to bark.

attributed to its thin cell walls, wide growth rings, and a lower proportion of latewood cells (Shmulsky and Jones 2019). Wood density can influence both retention and WPG values. Low-density wood typically contains larger and more open vessels, resulting in higher permeability compared to high-density wood (Amin *et al.* 2021). Consequently, the retention and WPG values in wood near the pith are higher than those in wood closer to the bark.

Surface Roughness

Surface roughness measurement is an important parameter in determining the quality of a product (Delima *et al.* 2022). Surface roughness quantified by the Ra parameter, defined as the arithmetic average of absolute deviations of the surface profile from its mean line (Hakim *et al.* 2017). Lower Ra values correspond to smoother surfaces, while higher Ra values signify greater surface irregularity (Tobing *et al.* 2024).

The Ra values in treated wood were higher than those in the untreated wood in both trees. This indicates that the wood surface became rougher after modification. The increase in surface roughness is suspected to be influenced by the use of citric acid during the impregnation process. This is supported by Reinprecht (2016), who stated that contact between wood and an acidic solution can lead to increased surface roughness. The study by Hanifah *et al.* (2025) showed that treatment using citric acid, a polar compound, tends to increase the surface roughness of wood. Chemical modification with citric acid shows that this compound contributes to the increased roughness of wood surfaces. These results are consistent with the study by Hanifah *et al.* (2023), which showed that Ra values increased significantly after furfurylation treatment. This indicates that the surface of furfurylated wood becomes rougher compared to untreated wood. Similar findings were also reported by Talai *et al.* (2016). The high acidity of the additive solution used in the furfurylation process likely causes the formation of microscopic cracks in the cell walls.

The type of sample cut used in this study, which was in the form of a radial section, also influenced the increase in Ra values. This is supported by Cahyono *et al*. (2022), who explained that heating using water or citric acid on radial sections led to increased Ra values, in some cases even doubling them. This roughness is caused by pressure from the tangential direction, which makes the surface rougher. The increase in roughness is associated with anatomical changes due to modification, such as fiber thinning and ray parenchyma deformation. Radial variation showed a fluctuating trend in Ra values for treated samples from both trees, whereas untreated samples tended to show a decrease from near the pith (A1 and C1) to the middle position (A5 and C5), followed by an increase toward the bark (A7 and C7) (Figure 3 and Figure 4). This variation in roughness values is likely due to differences in pore frequency and diameter at different radial positions from pith to bark. This finding is consistent with Lestari *et al.* (2016), who stated that pores with relatively large diameters can result in a less even or smooth surface. An increase surface roughness in this study implies greater material loss during the planing process. This is consistent with Korkut and Guller (2008), who stated that smoother wood produces less residue during planing and results in a higher-quality surface finish.

Surface Free Energy

Surface free energy (SFE) is an important variable in the wood finishing process. SFE can affect the wettability of wood (Wang *et al.* 2017). Figures 5 and Figure 6 show the SFE of untreated and treated wood.

The SFE value of untreated pine wood from the pith to the bark in sample A ranged from 38.60 to 42.49 mJ/m^2 and in sample C from 37.16 to 44.46 mJ/m^2. After treatment, the SFE values of sample A and sample C decreased dramatically, with values of 17.08–27.35 mJ/m2 and 16.87–23.34 mJ/m2, respectively. This decrease indicates that chemical modification with citric acid-glycerin causes the wood surface to become more hydrophobic, making it difficult for liquids to spread and penetrate into the wood. Tobing *et al.* (2024) show that the SFE value of Scotch pine wood treated with citric acid and glycerol decreased by 36.3% compared to the untreated wood. Higher SFE values indicate that the surface energy of the wood is greater, allowing liquids to spread and penetrate the wood more easily (Martha *et al.* 2020).

The SFE values of untreated and treated pine wood samples showed a radial increase from the pith to the bark. This is likely due to the extractive content in heartwood, making it more hydrophobic than sapwood. Heartwood is more hydrophobic because it has a higher extractive content (Martha *et al.* 2024). High extractive content can affect surface characteristics and compatibility with coatings (Jankowska *et al.* 2018). A decrease in surface free energy implies a reduction in the material's wettability, as solids with lower surface free energy are less easily wetted by liquids (Wang *et al.* 2017).

Wettability

The measurement of the K value of pine wood was carried out by calculating the change in contact angle that occurred in distilled water from the pith to the bark. The test results produced a graph showing a decrease in contact angle over time. Distilled water tends to spread slowly at the beginning, so the contact angle decreases gradually until it reaches a constant value. The data on the relationship between the contact angle of aquades from the pith to the bark is visualized in Figures 7 and 8.

The contact angle measurements show differences between the pith (C1) and bark (C7) regions, as shown

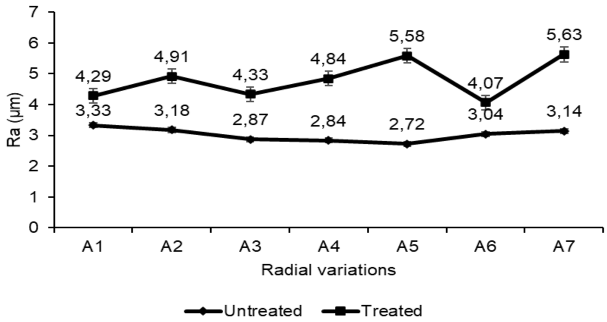


Figure 3 Surface roughness values of untreated and treated pine wood from pith (A1) to bark (A7) in tree A.

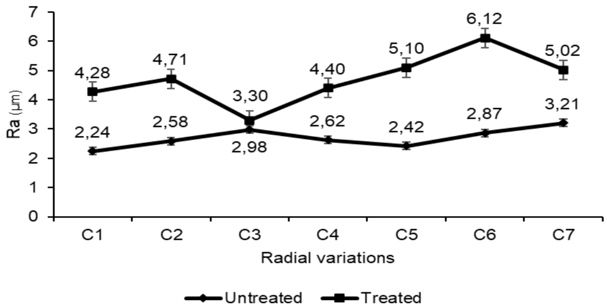


Figure 4 Surface roughness values of untreated and treated pine wood from pith (C1) to bark (C7) in tree C.

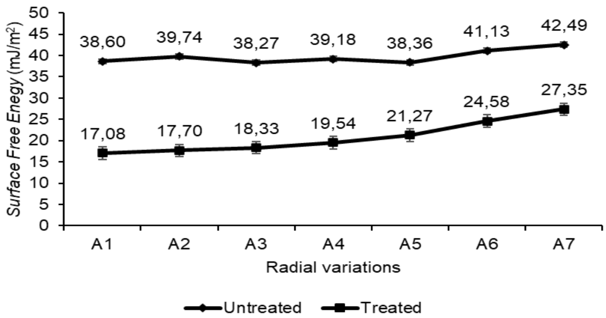


Figure 5 Total surface free energy values of untreated and treated pine wood from pith to bark on tree A.

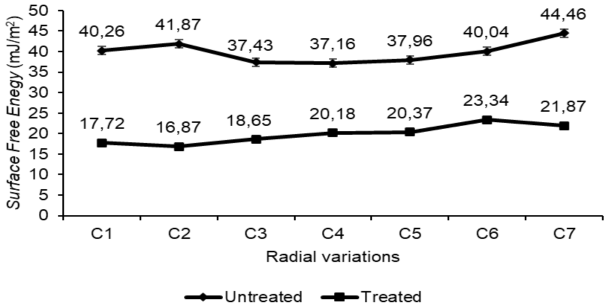


Figure 6 Total surface free energy values of untreated and treated pine wood from pith to bark on tree C.

in Figure 7. The higher contact angle values in the pith indicate stronger hydrophobic properties compared to the bark region. This is strongly suspected to be due to variations in wood anatomical structure, including differences in tracheid distribution and extractive compound concentration (Rowell 2012). A similar pattern can also be seen in Figure 8, where there is a decrease in contact angle from the pith to the bark. However, the rate of decrease in tree C is steeper than in tree A. This variation is thought to be caused by inter-individual variability in trees influenced by

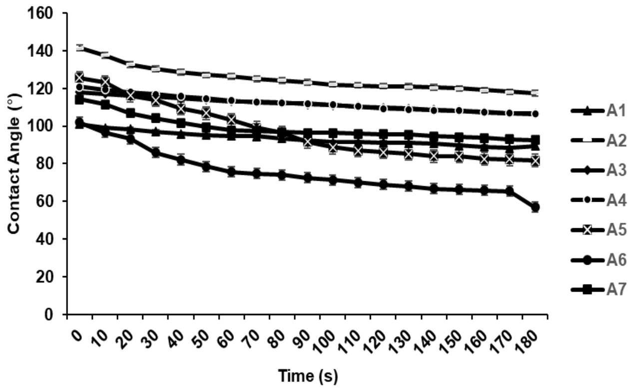


Figure 7 Changes in the contact angle of distilled water based on changes over time in treated pine wood from near the pith (A1) to near the bark (A7) on tree A.

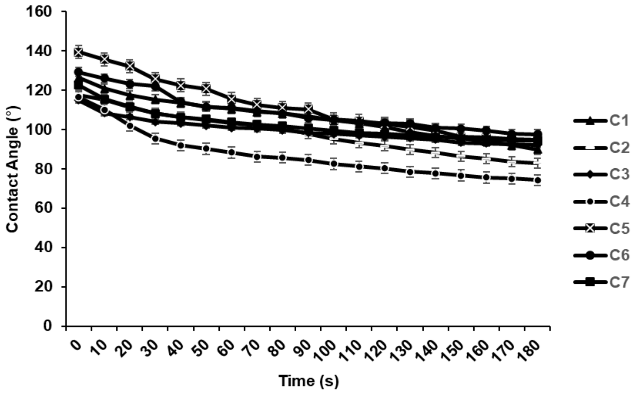


Figure 8 Changes in the contact angle of distilled water based on changes in time from treated pine wood near the pith (C1) to near the bark (C7) on tree C.

environmental or genetic factors (Esteves and Pereira 2009).

Contact angle measurement is one method for assessing the ability of wood to absorb liquids. The results of the study showed significant differences in K values between the two pine tree samples. Shown in Figure 9, in tree A the K value increases from 0.001 (near the pith) to 0.006 (near the bark), while in Figure 10 tree C shows a greater increase from 0.004 to 0.006. The average K values for trees A and C are 0.003 and 0.005, respectively. This data indicates that the wood of tree C has more hydrophilic properties than tree A, while the K values in the control treatment for both tree A and tree C show the same pattern, namely an increase from the pith to the bark. This pattern reflects radial differences in density, porosity, extractives, and surface roughness, which influence wood wettability (Tsoumis 1991). According to Abdussalam (2024), excessive nonpolar extractive content in wood can have a negative effect on wettability.

The part near the bark on both trees showed the highest K value, which was 0.006. This result was consistent with the untreated treatment, which showed the highest K value in the part near the bark. This was because the sapwood (near the bark) had higher moisture content than the heartwood (near the pith). According to Abdussalam (2024), this phenomenon is related to the higher Surface Free Energy (SFE) value in sapwood. Baldan (2012) research explains that a low SFE value results in a large contact angle (hydrophobic property), while a high SFE value results in a small contact angle (hydrophilic property). This mechanism is reinforced by Martha *et al.* (2020), who found that high SFE allows the surface energy of wood to break the surface tension of the liquid, thereby enhancing spreading and penetration capabilities. The pattern of increased wettability from the pith to the bark was observed in both trees. This is supported by Thygesen *et al.* (2010), who found that the bark region has more vessels and pores, enhancing water absorption capacity. The low K-value obtained in this study indicates that the treatment reduced the wood's wettability. This finding is consistent with the statement that a lower K-value corresponds to lower wettability (Shi and Gardner, 2001). Wettability determines the extent to which a liquid is drawn onto a material's surface (Marra 1992).

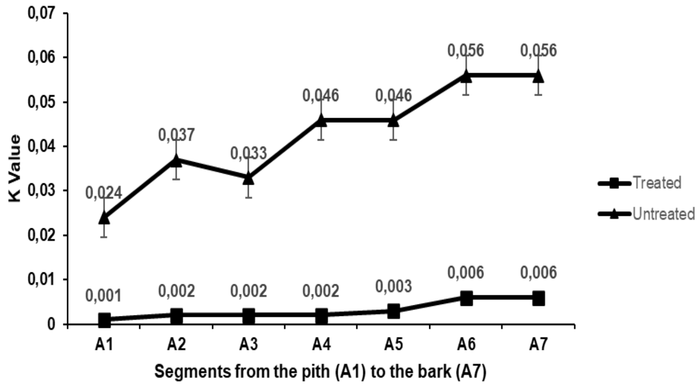


Figure 9 K values of treated pine wood from pith to bark on tree A.

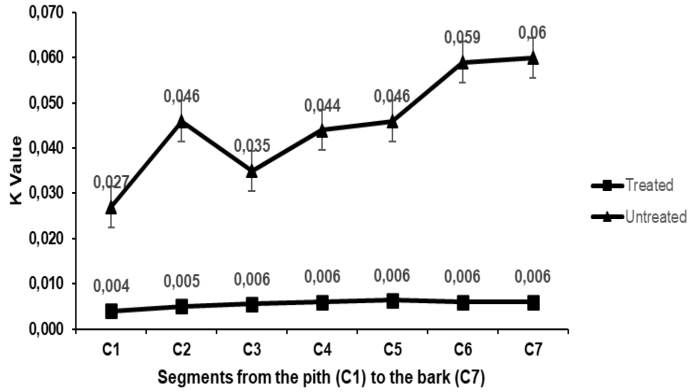


Figure 10 K values of treated pine wood from pith to bark in tree C.

CONCLUSION

Modification of pine wood using glycerin and citric a cid affects surface characteristics across radial variations. Retention, and WPG values increased, particularly near the pith, which has lower density. This treatment also increased surface roughness. In addition, surface free energy (SFE) significantly decreased, indicating that the wood surface became more hydrophobic and less wettable. Radial variations showed increasing values of SFE and wettability from pith to bark, reflecting structural differences in the wood. Glycerin–citric acid-treated pine wood had lower wettability compared to untreated wood. Overall, the surface characteristics of the treated pine wood across radial variations remained acceptable, with K values greater than 0, suggesting that the treated wood can still be coated using compatible paints.

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