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WETTABILITY OF CHEMICALLY AND THERMALLY MODIFIED OF FAST-GROWING TEAK WOOD

Keterbasahan Kayu Jati Cepat Tumbuh Termodifikasi Kimia dan Panas

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ABSTRACT

Fast-growing species are chemically and thermally modified to improve quality. These changes affect wood's chemical composition and surface characteristics. This study aims to analyze the durability of fast-growing teak wood that has been chemically and thermally modified. Chemical modification was carried out by impregnation using citric acid and polyethylene glycol (PEG) 400, while heat modification carried out at 150 °C. The wettability of wood was tested by measuring the contact angle with six types of liquid, aquades, toluene, glycerin, methanol, alkyd, and acrylic paint. The results showed that chemical modification treatment significantly improved the wettability of wood compared to control and heat treatment. Wood treated with citric acid and PEG 400 exhibits a lower contact angle, indicating increased liquid absorbency. Conversely, heat treatment increased the contact angle value making the wood more hydrophobic. Alkyd showed better wettability than acrylic paints, possibly due to their lower viscosity values. This suggests that chemical modification more effectively improves wood coating adhesion.

Keywords: citric acid; contact angle; PEG 400; wettability; wood modification.

ABSTRAK

Saat ini kayu fast growing species banyak dilakukan modifikasi secara kimia maupun panas untuk meningkatkan mutu kayu tersebut. Perlakuan modifikasi kimia maupun panas dapat mempengaruhi perubahan komposisi kimia terutama perubahan karakteristik permukaan. Penelitian ini bertujuan untuk menganalisis keterbasahan kayu jati cepat tumbuh yang telah dimodifikasi secara kimia dan panas. Modifikasi kimia dilakukan dengan impregnasi menggunakan asam sitrat dan polyethylene glycol (PEG) 400, sedangkan modifikasi panas dilakukan pada suhu 150 °C. Keterbasahan kayu diuji dengan mengukur sudut kontak dengan enam jenis cairan, yaitu akuades, toluena, gliserin, metanol, cat alkid, cat akrilik. Hasil penelitian menunjukkan bahwa perlakuan modifikasi kimia meningkatkan keterbasahan kayu secara signifikan dibandingkan kayu kontrol dan *heat treatment*. Kayu yang dimodifikasi dengan asam sitrat dan PEG 400 memiliki sudut kontak lebih rendah yang menunjukkan peningkatan daya serap terhadap cairan. Sebaliknya, *heat treatment* meningkatkan nilai sudut kontak sehingga kayu menjadi lebih hidrofobik. Cat alkid menunjukkan keterbasahan yang lebih baik dibandingkan cat akrilik, kemungkinan akibat nilai viskositasnya lebih rendah. Hal ini menunjukkan bahwa modifikasi kimia lebih efektif dalam meningkatkan keterbasahan kayu jati cepat tumbuh dan dapat meningkatkan kualitas daya rekat bahan pelapis kayu.

Kata kunci: asam sitrat; keterbasahan; modifikasi kayu; PEG 400; sudut kontak.

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A. INTRODUCTION

The rate of tree cutting in the forest is not balanced with its growth, so the forest area in Indonesia is decreasing rapidly from year to year (Halawane *et al.* 2011). Therefore, to compensate for the increasing demand for wood, fast-growing species from plantation forests are needed. Timber from plantation forests has a crucial role in meeting the raw material needs of the timber industry in Indonesia. Wood with the fast-growing species category generally has inferior properties such as small diameter, low quality, and high juvenile wood, so the yield is low (Rahayu *et al.* 2014). One of the woods in great demand and cultivated in plantations is teak (*Tectona grandis* Linn. F.)

The characteristics and basic properties of FGS teak wood are less durable and do not have striking patterns, so their appearance is less attractive (Darmawan *et al.* 2015). FGS wood has been chemically and thermally modified to improve the quality of the wood. Chemical and heat modification treatments can affect chemical composition changes, especially surface characteristics. Previous research has described wood modification using citric acid-sorbitol and heat treatment to improve wood surface properties, including wettability (Ishmah 2022). However, the study used pine wood and did not use PEG 400. Wood's wettability directly affects dimensional stability, adhesion, and resistance to wood-destroying microorganisms. In addition, chemical modification processes using citric acid and PEG 400 and heat modification can affect the wettability of wood by reducing its hygroscopicity.

Important parameters that can be used to characterize wood surfaces are surface roughness, free surface energy (SFE), and wettability (Qin *et al.* 2014). Surface roughness is essential to determine surface quality, especially in coating processes (Büyüksari *et al.* 2010). Darmawan *et al.* (2017) reported that surface roughness is crucial in determining wettability. The wettability of paint or coating material increases with increasing wood surface roughness (Darmawan *et al.* 2017). Previous research by Lestari *et al.* (2016) showed that surface roughness affects the wettability of wood. Therefore, it is necessary to research the effect of the level of wood surface roughness on wettability in chemically and heat-modified FGS teak wood.

B. METHODS

Materials and Tools

The material used was teak, a fast-growing species with a diameter of 38 cm. The chemicals used consisted of (citric acid (bratachem) and polyethylene glycol (PEG) 400 (bratachem)) for chemical modification, (distilled water, 50% methanol (Merck), toluene (Merck), glycerin (bratachem), acrylic paint with the trademark Propan Ultran Aqua politur Acrylic, and alkyd paint with the trademark Propan Ultran Lasur EL-501 Alkyd) for testing surface characteristics. The equipment used consisted of digital calipers, digital scales, an oven, a desiccator, a goblet, a measuring cup, an Erlenmeyer, a stirrer, aluminum foil, impregnation, surface roughness tester Mitutoyo type SJ-210, macro capture microscope, injection, ruler, cutter, tape, and camera.

Procedure

1. Preparation of Test Sample and Preparation of Impregnation Solution

Test samples were made with a size of 5 x 10 x 2 cm^3 (Figure 1). All test samples were taken from sapwood. The treatments were control, chemical modification through impregnation using citric acid, polyethylene glycol (PEG) 400, and heat modification. Each solution used a concentration of 40% for citric acid and 50% for PEG diluted with distilled water. All test samples were not sanded before and after treatment to equalize the wood surface.

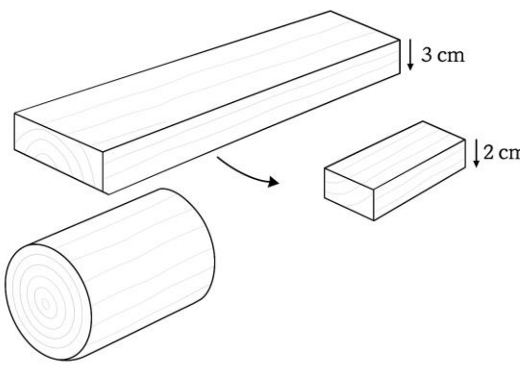

Figure 1. Cutting the test sample

2. Chemical Modification Through Impregnation

The sawn test samples were dried at 103±2 ºC for 48 hours to determine the initial mass (m_0) and volume (v_0). The samples were then immersed with the impregnation solution in an impregnation tube under vacuum at 0.5 bar for 15 minutes, followed by pressurization at 2.5 bar for 1 hour. The test samples were then drained and wrapped in aluminum foil. Then, the test samples were placed in an oven at 60°C for 48 hours. All oven-modified test samples were measured for mass (m_1) and volume (v_1).

3. Heat Modification

The heat modification method refers to the research of Martha *et al.* (2021) and Hanifah (2022). The sawn test sample was heat treated at 150 ºC. Before being heat treated, the test sample's mass (m_0) and volume (v_0)were measured. Heat treatment was carried out based on the following stages: (1) heating to 100 ºC for 1 hour, (2) increasing the temperature from 100 ºC to a final temperature of 150 ºC, (3) heating at 150 ºC for 2 hours, and (4) the oven was turned off, and the test sample was left for 1 hour in the oven. After the heat treatment, the test samples were weighed for mass (m_1), and volume (v_1) was measured.

4. Surface Roughness Measurement

The surface roughness of the test samples was measured using a Mitutoyo SJ-210 tester. Measurements were taken following the direction perpendicular to the fibers at five different points on the wood surface to obtain roughness values representative of all parts and ensure accuracy and consistency. Wood surface to obtain roughness values representative of all parts. Measurements based on ISO 4287 standard: 1997 (ISO 1997) using a cut-off length of 0.8 mm, a measurement path length of 6 mm, and a 0.5 mm/second measurement speed. The surface roughness measurement parameter used is arithmetical mean roughness (Ra), referring to JIS B 0601:2001.

5. Contact Angle Measurement

The dynamic contact angles of standard test liquids (distilled water, toluene, 50% methanol, glycerin) were used for SFE measurements. In contrast, alkyd and acrylic coating materials were used to determine wettability values. The wood sample was placed on a flat table parallel to the camera during the measurement. Liquids and paints were dripped through an injection using the screw method to obtain the same drip volume (0.02 ml). For each surface of the test sample, the contact angle was measured at 3 points, and the repetition of 3 points met the statistical repeatability to reduce the experimental error with the size of the test sample with a small surface. The dripping results were recorded for 180 seconds. Each video recording was cut using GOM Player software with a cutting interval of 10 seconds in each solution, except for the fast-absorbing solution using an interval of 0.1 seconds for 19 images.

The contact angle of each image slice was then measured using Image-J 1.46 with dropsnake plugin analysis. Contact angles were measured on both sides of the droplet and then averaged. As a result of image cropping, 19 points of each droplet were recorded to obtain a contact angle curve against time. The contact angle measurements were carried out in a room with a temperature of 23±2 °C and a relative humidity of 80±5%.

6. Determination of Equilibrium Contact Angle and Wettability Value

Determination of wettability and SFE values is done by measuring the constant contact angle between the liquid or paint and the wood surface. The constant contact angle value was determined based on a segmented regression equation between time (x) and contact angle (y) using the PROC NLIN program from SAS. Then, the K-value of the S/G model (Shi & Gardener 2001) was determined using the XLSTAT software. The S/G model uses the following equation:

$$\theta = \frac{\theta i.\theta e}{\theta i+(\theta e-\theta i)\exp\left[K\left(\frac{\theta e}{\theta e-\theta i}\right)\right]t} \qquad (1)$$

Where, θ is the contact angle at any given time, θi is the initial contact angle, θe is the equilibrium contact angle, t is the time, and K is the constant for the contact angle change rate.

7. Determination of Surface Free Energy (SFE)

Surface free energy (SFE) is one of the thermodynamic quantities that describe the state of atomic equilibrium in the surface layer of a material (Żenkiewicz 2007). Surface energy is a combination of dispersed (non-polar) energy and polar energy. The basis for determining the solid surface free energy (γs) is the size of the contact angle (θ) of a standard liquid droplet (with known γl) on the plane surface of the test sample. The surface tension of a liquid is measured or evaluated from the contact angle on a known solid surface. Currently, many methods are used to determine SFE values. The two-liquid method was modified into a multi-liquid method to assess the SFE value and its components proposed by Rabel (1971) by using a regression line with the following equation:

$$(1 + \cos\theta)\frac{\gamma l}{(\gamma_l^d)^{1/2}} = \left(\gamma_s^d\right)^{1/2} + \left(\gamma_s^p\right)^{1/2}\left(\frac{\gamma_l^p}{\gamma_l^d}\right)^{1/2} \qquad (2)$$

Where θ is the equilibrium contact angle, γ_l is the free energy of the liquid, γ_l^d is the dispersion component of the liquid, γ_s^d is the surface energy of the solid that belongs only to the dispersion component, γ_l^p is the polar component of the liquid, and γ_s^p is the surface energy of the solid that belongs only to the polar component.

Table 1. Surface tension values of the test liquid surface and its components (in $mJ.m^{-2}$) used for wood surface free energy calculations (Yuningsih *et al.* 2019).

Standard test liquid	γl^p	γl^d	γl
Distilled water	21.8	51.0	72.8
Methanol	12.9	22.7	35.6
Toluene	2.3	26.1	28.4
Glycerin	30.0	34.0	64.0

Description: γl^p = Value of polar component of surface tension, γl^d = Value of dispersion component of surface tension, γl = Value of total surface tension.

Data Analysis

Data results were analyzed using analysis of variance (ANOVA) of a complete randomized factorial experiment. The mean difference test used Duncan's test at α = 5%. The equation model used is as follows:

$$Y_{ijk} = \mu + A_i + E_{ijk} \qquad (3)$$

Where, Y_{ijk} is the observation value of the i treatment and j replication, μ is the general average, A_i is the effect of i treatment (control, chemical modification of citric acid, PEG, and heat modification), and E_{ijk} is the residual effect.

C. RESULTS AND DISCUSSION

Surface Roughness

This study on the effect of chemical and heat treatment on treatment roughness showed significant modification of wood surface characteristics. The Ra value shows that heat treatment at 150 ºC produces a surface roughness value (Ra) of 15.29 µm, which is the lowest value compared to other chemical treatments, such as citric acid (18.07 µm) and PEG 400 (15.80 µm), as well as the control (16.52 µm). It shows that heat treatment can refine the surface of wood, possibly due to the evaporation of volatile substances and recrystallization of chemical compounds in wood during heating, resulting in a more homogeneous and flat surface (Hanifah *et al.* 2022). This statement is in line with the research of Fendi *et al.* (2017), which showed that heat treatment can change the anatomical structure of wood, reduce the degree of crystallinity, and reduce surface roughness. Therefore, treatment at 150 ºC can potentially improve the surface quality of FGS teak wood for applications requiring a low roughness level.

Table 2. Surface roughness values of teak wood

Treatment	Surface Roughness (µm)		
	Rz	Ra	Rq
Control	84.83 ± 11.83^{ab}	16.52 ± 2.24^{ab}	20.41 ± 2.91^{ab}
Heat treatment	79.42 ± 8.76^{a}	15.29 ± 2.38^{a}	18.72 ± 2.59^{a}
Citric acid	90.34 ± 12.29^{b}	18.07 ± 2.34^{b}	22.14 ± 2.95^{b}
PEG 400	81.99 ± 10.37^{ab}	15.80 ± 1.58^{a}	19.63 ± 2.22^{ab}

Description: Rz = ten-point height, Ra = arithmetical mean roughness, Rq = root mean square roughness. a-b values followed by the same letters do not differ significantly (α = 0.05) based on the Duncan test.

The effect of citric acid treatment, which is a polar compound, tends to increase the roughness of the wood surface. It may increase the ability of the wood to interact with the liquid or coating material, which is expected to improve the adhesion of the coating material to the wood. Although PEG 400 treatment has a lower polarity compared to citric acid, it can reduce the roughness of the wood surface, thereby reducing wettability and the adhesion of the coating material. Chemical modification using citric acid and PEG 400 showed that citric acid tends to increase wood surface roughness, while PEG 400 reduces wood surface roughness (Tobing *et al.* 2024).

Surface Free Energy (SFE)

The total SFE, polar and dispersive components of control, heat treated, citric acid, and PEG 400 teak wood are presented in Figure 2. The total SFE and its components exceeded the control or untreated wood. The highest surface free energy was obtained by PEG 400-impregnated teak wood with a value of 67.99 $mJ.m^{-2}$, followed by heat treatment at 40.04 $mJ.m^{-2}$, citric acid-impregnated teak wood at 39.42 $mJ.m^{-2}$, and the lowest was control teak wood at 29.39 $mJ.m^{-2}$. It shows that although PEG 400 has a lower polarity, it can significantly increase the SFE of teak wood, which is higher than that of citric acid. The increase in the total SFE value could be due to the decrease in hydrophobicity after the thermal and chemical modification process.

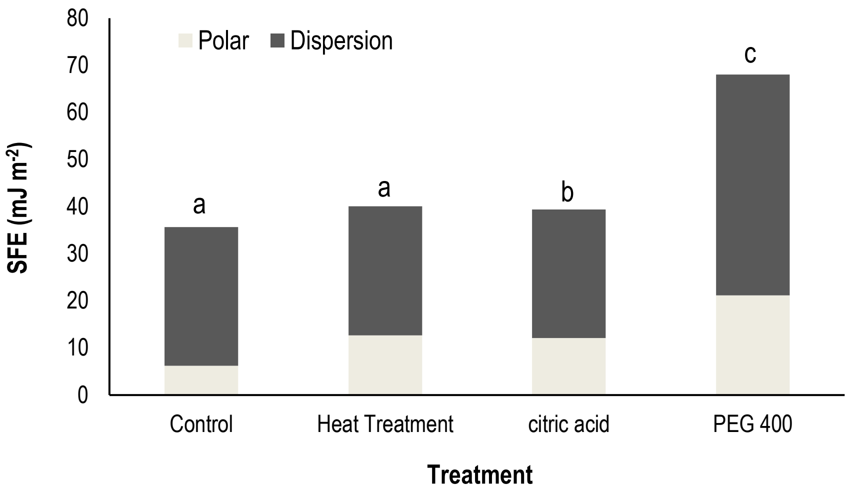

Figure 2. Teak wood surface free energy results. Note: a-c values followed by the same letters do not differ significantly (α = 0.05) based on the Duncan test.

Citric acid, as a polar compound, can increase the SFE value by increasing the polarity of the wood surface, which increases the ability of wood to interact with liquids or other materials (Lee *et al.* 2014). It is in accordance with previous research showing that polar compounds in the material have a higher ability to bind other components, thus increasing surface free energy (Kúdela 2014; Aliofkhazraei 2015). It causes an increase in surface-free energy in wood impregnated with polar compounds such as citric acid (Namazi & Adeli 2005; Chandra & Karak 2018). In contrast, although PEG 400 has a low polarity, treatment with PEG 400 still significantly increased SFE values. However, the increase is less than citric acid (Wilder *et al.* 2003).

The results of the increase in surface free energy after heat treatment are not in line with several other studies that show a decrease in energy after treatment (Gerardin *et al.* 2007; Santos & Gonçalves 2016). Heat treatment can degrade hemicellulose, reducing free polar components, especially hydroxyl groups in wood (Dubey *et al.* 2012). The heat treatment in this study possibly degraded part of the wood surface, thus reducing the surface area. Reducing wood surface area can increase surface free energy in calculations (Gindl *et al.* 2001). Therefore, the heat treatment in this study may improve the surface-free energy of the wood produced.

Contact Angle

The equilibrium contact angles (θ_e) of untreated teak wood, heat treatment, PEG, and citric acid are presented in Table 3. The equilibrium contact angle value is an essential factor in determining the wettability value of the liquid or coating material on the wood surface (Martha 2021). The results showed that some contact angle values on control teak wood were higher, indicating that the teak wood surface was more hydrophobic than wood treated with chemicals or heat. However, after chemical and heat treatment, there was a significant change in the contact angle value of the wood. The contact angle value of acrylic paint heating treatment is higher than that of alkyd paint. Following Martha's study (2019), the contact angle of teak wood heat treatment with acrylic paint was 37.09, and the alkyd paint was 10.14. The contact angle decreased along with the length of wetting of the paint on the wood surface. Alkyd paint showed a lower contact angle than acrylic paint. This is due to the lower surface tension of the alkyd paint layer than acrylic paint.

Table 3. Equilibrium contact angle values in teak wood

Treatment	θ_e Distilled water	θ_e Toluene	θ_e Glycerin	θ_e Methanol	θ_e Alkyd	θ_e Acrylic
Control	35.65^{b}	13.85^{a}	76.19^{a}	10.01^{a}	48.75^{c}	26.86^{c}
Heat treatment	25.95^{a}	0.10^{c}	78.17^{a}	0.76^{c}	0.30^{a}	56.11^{b}
Citric acid	28.73^{ab}	5.46^{b}	72.73^{ab}	6.64^{b}	3.46^{b}	76.40^{ab}
PEG 400	26.76^{a}	3.57^{b}	33.10^{b}	18.44^{ab}	0.24^{a}	84.63^{a}

Note: a-c values followed by the same letters do not differ significantly (α = 0.05) based on the Duncan test.

Higher surface roughness usually increases the surface area that can interact with the liquid, thus affecting the contact angle between the wood surface and the liquid. The contact angle values of heat-treated teak wood showed decreased contact angle values in distilled water, toluene, and methanol. The value of Ra heat treatment decreased when the wood surface became smoother, but the contact angle decreased. It could be due to chemical changes in the wood surface during heat treatment, so the wood becomes more hydrophobic, increasing the contact angle in some liquids, such as glycerin (78.17).

The highest contact angle values of acrylic paint in PEG 400 and citric acid modification were 76.40 and 84.63, respectively. It is suspected that citric acid-treated teak wood affects the hydrophilicity of the wood. Although citric acid and PEG 400 chemical treatments have higher Ra values than heat treatments, both treatments increase the interaction of the wood surface with the liquid. Shao *et al.* (2019) reported that citric acid is proven to make wood more hydrophobic, so citric acid-modified wood has water-repellent properties, increasing contact angle. In contrast, the toluene solvent produced lower contact angle values in all treatments. The toluene solution is suspected to have a faster absorption capacity on the wood surface than other solutions or coating materials.

Wettability

The K value (wettability) describes how fast a liquid can spread and penetrate into the wood surface (Gray 1962). Measuring the wettability of the paint liquid on the wood surface is vital. A K=0 value indicates very poor wettability. The K value of wood is an essential factor in measuring the wettability of a liquid or coating material on a wood surface. A higher K value indicates better wettability. Darmawan *et al.* (2017) stated that the greater the K value, the faster the time required to reach the equilibrium contact angle and the quicker the paint can spread on the wood surface.

The K values of acrylic paints on control, thermally modified, PEG and citric acid-impregnated teak wood were 0.32, 0.05, and 0.57, respectively, while those of alkyd paints were 7.80, 5.45, 76.40, and 100.39 (Figure 3). The K value in various treatments shows that the highest value in acrylic paint is in citric acid, PEG, control, and thermal modification. These results are the same as the wettability value of alkyd paints.

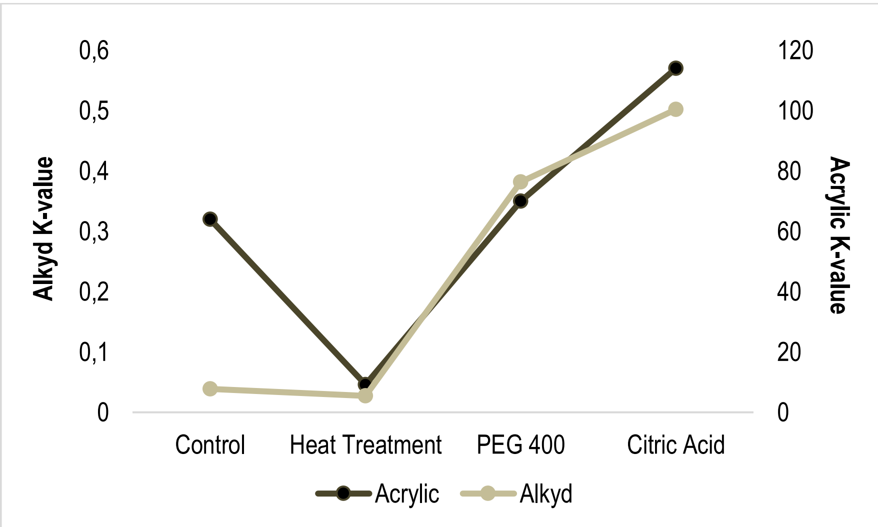

Figure 3. K-value of teak wood

The K value of acrylic paint and alkyd paint increased after being treated with chemical modification of citric acid and PEG 400, while it decreased after being heat treated. It can be caused by the hydrophilic nature of the wood chemically modified with citric acid and PEG, which has higher water-absorbing properties than the control wood. The K value decreases as the heating temperature increases. The wettability value is influenced by the macroscopic characteristics of wood, such as density, extractive substances, surface roughness, and moisture content (Unsal *et al.* 2011). Chemical modification treatment of citric acid and PEG 400 increased the wettability of teak wood significantly compared to control and heat treatment. Thus, heat treatment is less effective in improving the absorbency of paint or liquid on wood.

The viscosity of the liquid can affect the wettability of the wood. Alkyd paints have better wettability compared to acrylic paints because the viscosity of alkyd paints is lower. This is due to the lower viscosity of alkyd paint compared to acrylic paint. The wettability of the wood surface will decrease as the viscosity of the liquid increases (Monni., 2007). Therefore, citric acid and PEG 400 can increase the wettability of teak wood, and alkyd paints still have better absorbency than acrylic paints on chemically treated wood surfaces.

D. CONCLUSION

Chemical modification treatment using citric acid and PEG 400 increased the wettability of fast-growing teak wood compared to the control wood. In contrast, heat treatment tends to reduce the wettability of wood. This result is shown through an increase in SFE value and a decrease in contact angle in chemically modified wood, and an increase in the ability of wood wettability properties. In addition, alkyd paints have better wettability than acrylic paints, likely due to their lower viscosity. Factors such as surface roughness, chemical composition of the wood, and polarity of the compounds contained in the modification materials play a role in determining the wettability of fast-growing teak wood.

AUTHOR'S DECLARATION

- Conflicts of Interest: None.
- We here by confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for republication,which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- No potentially identified images or data are present in the manuscript.

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