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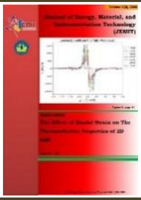

Effect of Corona Plasma Radiation on the Contact Angle and Wettability of Bamboo Surface

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Abstract

Bamboo is a widely utilized non-timber forest product, often innovated into laminated boards, particle boards, and plywood. The outer bamboo surface (skin) possesses high compressive strength but exhibits hydrophobic properties, limiting its wettability toward liquids. Plasma corona technology, operating at atmospheric pressure and using surrounding gases at a relatively low cost, offers a promising solution to modify bamboo surfaces by increasing their surface energy. This study aimed to analyze the hydrophilic resistance and wettability of bamboo surfaces treated with plasma corona, measured through contact angle analysis. The direct drop method was employed by dripping water and phenol formaldehyde (PF) fluids onto bamboo surfaces treated with plasma corona. Contact angle measurements were performed using ImageJ drop analysis software. Results demonstrated that plasma corona treatment significantly reduced contact angle values, improving surface wettability. Untreated bamboo surfaces showed contact angles of 72.7° for water and 111.5° for PF fluids, indicating limited wettability. Post-treatment, the contact angles decreased to less than 30° for water and below 90° for PF, signifying enhanced wettability. However, the hydrophilic properties were found to be non-permanent, with contact angle values gradually increasing over 13 days of observation. Among the tested parameters, plasma corona treatment with a current strength of 75 mA for 5 minutes provided the most optimal wettability improvement for water and PF fluids. This study highlights the effectiveness of plasma corona in enhancing bamboo surface wettability, making it more suitable for applications requiring adhesive bonding or fluid spreading in industrial processes.

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Abstrak

Bambu merupakan salah satu hasil hutan bukan kayu yang banyak dimanfaatkan, sering diolah menjadi papan laminasi, papan partikel, dan kayu lapis. Permukaan luar bambu (kulit) memiliki kekuatan tekan yang tinggi, tetapi bersifat hidrofobik, sehingga membatasi keterbasahannya terhadap cairan. Teknologi plasma corona, yang bekerja pada tekanan atmosfer dengan menggunakan gas di lingkungan sekitar dan biaya yang relatif rendah, menjadi solusi untuk memodifikasi permukaan bambu dengan meningkatkan energi permukaannya. Penelitian ini bertujuan menganalisis ketahanan sifat hidrofilik dan keterbasahan permukaan bambu yang telah diberi perlakuan plasma corona dengan mengukur sudut kontak. Metode tetesan langsung digunakan dengan meneteskan air dan cairan phenol formaldehyde (PF) pada permukaan bambu yang telah diberikan plasma corona, diukur menggunakan perangkat lunak ImageJ. Hasil penelitian menunjukkan bahwa perlakuan plasma corona mampu menurunkan nilai sudut kontak secara signifikan, meningkatkan keterbasahan permukaan bambu. Permukaan bambu tanpa perlakuan memiliki sudut kontak sebesar 72,7° untuk air dan 111,5° untuk PF, yang menandakan keterbasahan terbatas. Setelah diberi plasma corona, sudut kontak menurun

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menjadi kurang dari 30° untuk air dan di bawah 90° untuk PF, menunjukkan peningkatan keterbasahan. Namun, sifat hidrofilik ini tidak bersifat permanen, dengan nilai sudut kontak yang perlahan meningkat selama 13 hari pengamatan. Perlakuan plasma corona paling optimal ditemukan pada kuat arus 75 mA dengan waktu perlakuan 5 menit, menghasilkan peningkatan keterbasahan terbaik untuk air dan PF. Penelitian ini membuktikan efektivitas plasma corona dalam meningkatkan keterbasahan permukaan bambu, sehingga lebih cocok untuk aplikasi industri yang membutuhkan daya rekat atau penyebaran cairan yang baik.

1. Introduction

The increasing human population increases the need for wood timber materials in the construction and industrial sectors (Nourbaksh et al., 2008; Dukarska et al., 2017). As a result, the timber supply is increasingly depleted, which has impacted the increasing rate of natural forest degradation in Indonesia (Kementerian Lingkungan Hidup dan Kehutanan, 2016). The abundant types and quantities of bamboo in Indonesia and the rapid growth rate of bamboo make it a frequently used alternative (Clark et al., 2015; Hastuti et al., 2018). The superior characteristics of bamboo, such as high tensile strength, lightweight, and flexibility, allow bamboo to be used in various applications. One technology for processing bamboo into high-strength wood-like blocks is making biocomposites or laminated boards (Ismanto et al., 2019; Ameh et al., 2021). In the development of technology-based products, adhesives are an essential element, where phenol formaldehyde (PF) dominates as an adhesive with a usage rate reaching 95% (Priyanto & Yasin, 2019; Dewi et al., 2023). Laminated boards are generally made using the middle part of bamboo without skin because they interfere with gluing (Agustina et al., 2015; Dayadi et al., 2017).

One of the challenges in utilizing bamboo is the hydrophobic nature of its outer skin, which inhibits the adhesive strength on the surface (Borowski et al., 2022). As a result, the outer part of bamboo is often not utilized in the production process, even though it has higher strength and durability than the inner part (Budi, 2007; Ndale, 2013). If the outer surface properties of bamboo can be modified, its potential for utilization will increase significantly, especially in the composite industry. One promising approach to overcome this problem is to use plasma radiation.

Plasma radiation is an environmentally friendly surface modification method because it does not involve hazardous chemicals (Hidayanti, 2021). Plasma is an ionized gas containing electrons, ions, and free radicals, which provide unique characteristics and distinguish it from ordinary gases (Harianingsih et al., 2023). Corona plasma is an economical choice for bamboo surface modification among the various types of plasma. The corona plasma process can be carried out at atmospheric pressure and room temperature, making it simpler and more efficient for industrial purposes compared to other types of plasma, such as glow discharge or arc plasma (Li et al., 2019; Wang & Cheng, 2020).

Previous studies have shown that plasma radiation can increase the hydrophilic properties of bamboo surfaces, thereby increasing their adsorption capacity to adhesives (Guo et al., 2021; Putra & Susanto, 2021). However, these hydrophilic properties tend to be impermanent, where the surface contact angle can return to its initial condition within a certain period, for example, 2 to 5 days after treatment (Lopes et al., 2018; Wang & Cheng, 2020). Therefore, it is important to understand the duration of the resulting hydrophilic properties, especially to ensure the sustainability of the performance of bamboo-based products.

This study uses Indonesian bamboo samples treated with corona plasma with variations in radiation time and current strength. The survey scope includes contact angle measurements to analyze the wettability of bamboo surfaces to water and phenol formaldehyde (PF) fluids, as well as changes in contact angles that reflect the resistance of surface hydrophilic properties after plasma treatment. In addition, this study also evaluates the duration of plasma treatment effectiveness on the hydrophilic properties of bamboo surfaces, which is relevant to the waiting time before the material enters the advanced production stage. The results of this study are expected to contribute to improving the efficiency and quality of bamboo utilization in the industry.

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2. Research Methods

This research was conducted at Institut Teknologi Sumatera, with a scope of activities including viscosity measurement at the Agricultural Industrial Technology Laboratory, solids content measurement at the Forestry Engineering Laboratory, and contact angle analysis at the Instrumentation and Automation Engineering Laboratory. The research flow in this study is shown in **Figure 1**.

2.1 Materials and Method

This study used various tools that were adjusted to the testing needs. Solid content was measured using an oven, digital scales, desiccators, and aluminum foil. Viscosity measurements were carried out using an NDJ 5SS viscometer and a beaker. Contact angle analysis was carried out using a syringe for liquid dripping, a digital camera for recording, and VLC Media Player and ImageJ software with the Drop Analysis-DropSnake plugin for measurement. Test result data were analyzed statistically using SPSS software.

The materials used include bamboo samples measuring 3 × 1 × 0.5 cm that have previously been treated with corona plasma with variations in radiation time (1, 3, and 5 minutes) at current strengths of 50 mA and 75 mA, as well as control samples without treatment. In addition, distilled water with a viscosity of 1.0 cps and regular-type phenol formaldehyde (PF) with a viscosity of 100–170 cps from PT Dover Chemical were also used as test materials.

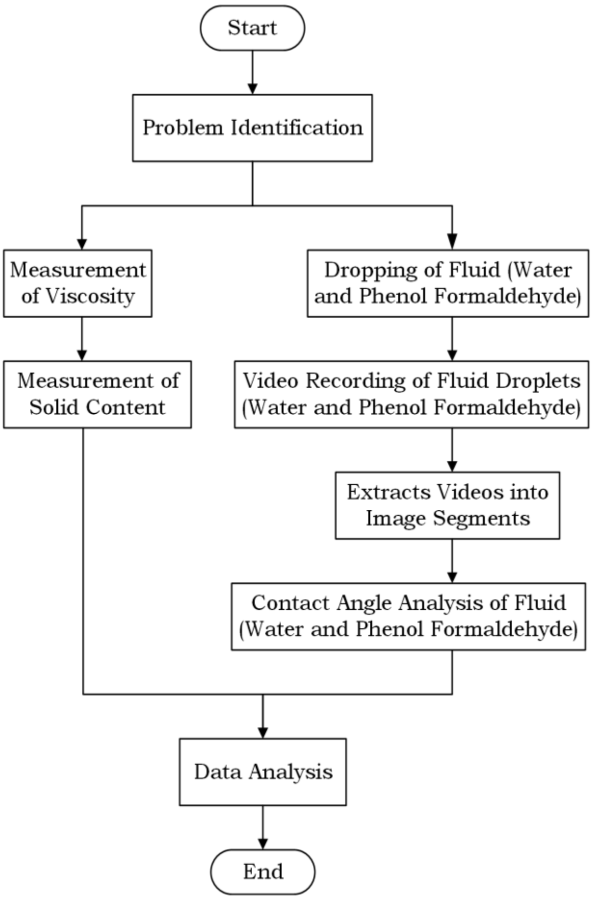


Figure 1. Research flowchart

2.2 Solid Content Measurement

A total of ±3 g of PF was weighed, then heated in an oven at 103 °C for 24 hours until it reached a constant mass and cooled in a desiccator. The solid content was calculated using **Equation 1** (Bahar, 2019),

$$SC\ (\%) = \frac{C\ (g) - A\ (g)}{B\ (g)} \times 100\% \quad (1)$$

Where SC (%) = Solid content (%), A = mass of container (g), B = Mass of sample before drying (g), and C = Mass of container and sample after drying (g).

2.3 Contact Angle Measurement

Liquid droplets were applied to the bamboo surface to measure fluid interaction with the surface using the sessile drop method. The droplet recordings were analyzed periodically (starting from 0 hours to day 13) to measure changes in contact angle using ImageJ (Panggugah, 2021). Variations in plasma radiation time (0, 1, 3, and 5 minutes) and current strength (50 mA and 75 mA) were used to evaluate their effects on the hydrophilic properties of bamboo.

2.4 Data Analysis

Statistical analysis was performed using ANOVA and Independent Sample t-test on SPSS software with a significance level (α) of 0.05. ANOVA was used to compare the variation of plasma radiation time, while t-test was used to compare the differences in the variation of plasma current strength (50 mA and 75 mA). Significant differences are indicated by p-value <0.05.

3. Results and Discussions

3.1 Contact Angle Analysis

1. Contact Angle of Water. The contact angle value of water fluid on the bamboo surface without plasma corona treatment is 72.7°, as shown in **Figure 2**, indicating that the bamboo surface is partially wettable because the contact angle is in the range of 30°-90°. This is due to the low viscosity of water (1.0 cPs) so that water tends to spread faster on the surface (Kwok & Neumann, 2000; Wang & Cheng, 2020).

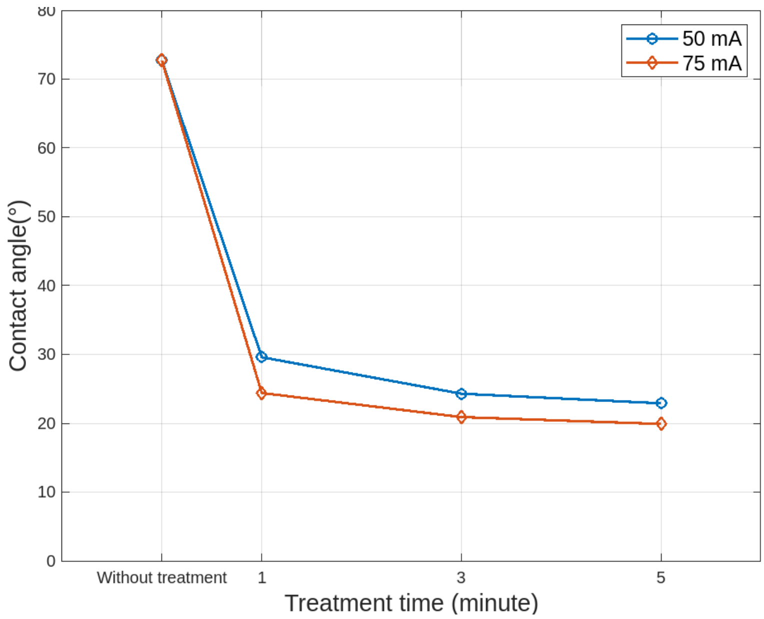


Figure 2. Contact angle of water with plasma treatments

After plasma corona treatment, at a current strength of 50 mA, the contact angle values decreased to 29.6° (1 minute), 24.3° (3 minutes), and 22.9° (5 minutes); At a current strength of 75 mA, the contact angle values became 24.4° (1 minute), 20.9° (3 minutes), and 19.9° (5 minutes). This significant decrease in the contact angle indicates that the bamboo surface becomes hydrophilic (contact angle <30°) after plasma corona treatment. This reduction in contact angle is comparable to that observed in bamboo subjected to glow-discharge treatment, where the water droplet contact angle decreased from 115° to 14° (Wang & Cheng, 2020).

The current strength and duration of plasma treatment greatly affect the degree of decrease in the contact angle value. Plasma corona with a current strength of 75 mA and a time of 5 minutes showed better results in increasing hydrophilic properties than a current strength of 50 mA. This is because a higher current strength produces more ions, electrons, and active radicals that can modify the bamboo surface more effectively (Lopes et al., 2018; Putra & Susanto, 2021).

2. Contact Angle of Phenol-Formaldehyde (PF). The contact angle value of PF fluid on the bamboo surface without plasma treatment was 111.5°, as shown in **Figure 3**, indicating hydrophobic properties (contact angle >90°). This is due to the high viscosity of PF (100-170 cPs), so the liquid tends to be difficult to spread on the surface (Aksoy et al., 2022).

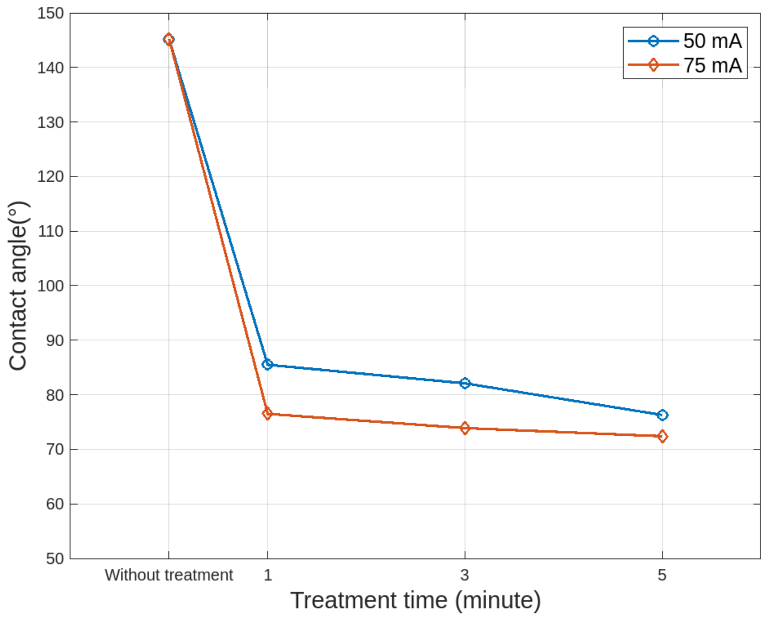


Figure 3. Contact angle of PF with plasma treatments

After corona plasma treatment, at a current strength of 50 mA, the contact angle values decreased to 85.5° (1 minute), 82.1° (3 minutes), and 76.3° (5 minutes); At a current strength of 75 mA, the contact angle values became 76.5° (1 minute), 73.9° (3 minutes), and 72.4° (5 minutes). This decrease in the contact angle value indicates that the bamboo surface, which was initially hydrophobic, changed to partially wet (contact angle 30°-90°) after plasma treatment. As with water fluid, plasma treatment with a current strength of 75 mA and a time of 5 minutes gave better results than the 50 mA treatment because the greater plasma energy increased the surface wettability more significantly. This variation is comparable to the bamboo treated with cold oxygen plasma, assessed using phenol-formaldehyde resin, where the contact angle reduced from 104° to 52° (Wang & Cheng, 2020).

The results showed that the current strength and duration of the corona plasma treatment significantly affected the bamboo surface's wettability properties. Higher current strength (75 mA) produced more plasma energy capable of breaking down the molecular structure of the bamboo surface, creating polar groups that increased the hydrophilic properties (Li et al., 2019; Putra & Susanto, 2021). In addition, longer treatment times provided a greater opportunity for the plasma to modify the surface evenly, thereby significantly reducing the contact angle.

3.2 Hydrophilic Properties of Bamboo Surface Resilience After Corona Plasma Treatment

In **Figure 4**, the contact angle of water fluid decreased drastically immediately after plasma corona treatment (day 0), indicating that the bamboo surface became highly hydrophilic. The initial contact angle values on day 0 varied based on the current strength and treatment time, namely: 50 mA: 30.6° (1 min), 24.3° (3 min), and 22.9° (5 min); 75 mA: 24.4° (1 min), 20.9° (3 min), and 19.9° (5 min). However, over time (until day 13), the contact angle values increased, indicating that the hydrophilic nature of the surface was not permanent. On day 13: 50 mA: 39.6° (1 min), 33.0° (3 min), and 29.3° (5 min); 75 mA: 34.1° (1 min), 28.6° (3 min), and 28.5° (5 min).

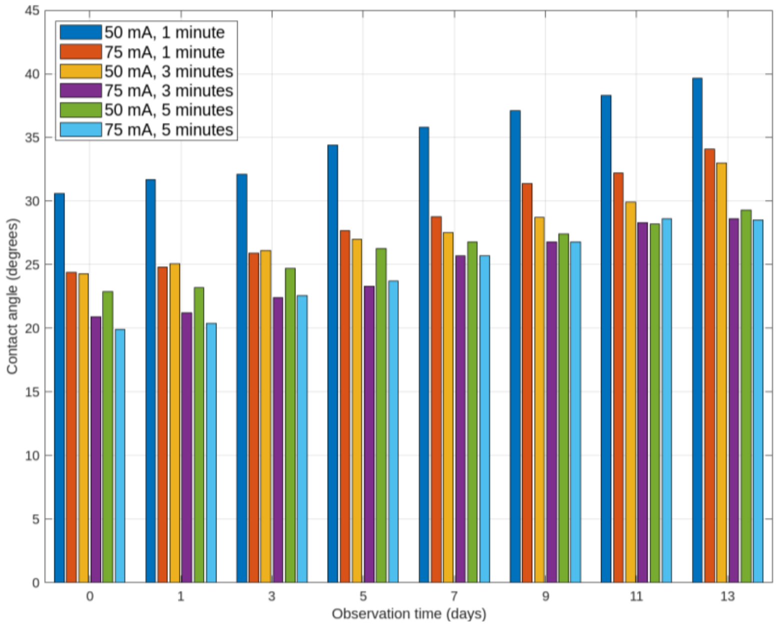


Figure 4. Contact angle of water after plasma treatments

This increase indicates molecular recombination on the material surface, where polar molecules produced during plasma treatment slowly return to their original state when exposed to oxygen, moisture, or the surrounding environment (Volpe et al., 1994; Altgen et al., 2020). In addition, the length of treatment time and plasma current strength affect the initial level of hydrophilicity and its durability. Treatment with 75 mA for 5 minutes resulted in the best hydrophilic durability compared to other treatments.

In **Figure 5**, the contact angles of PF fluids also showed a significant decrease immediately after corona plasma treatment (day 0): 50 mA: 85.5° (1 min), 82.1° (3 min), and 76.3° (5 min); 75 mA: 76.5° (1 min), 73.9° (3 min), and 72.4° (5 min). However, as with water fluids, the contact angles of PF increased gradually during the 13 days of observation, reflecting the impermanent hydrophilic nature. On day 13: 50 mA: 123.3° (1 min), 124.1° (3 min), and 115.5° (5 min). 75 mA: 120.6° (1 min), 112.4° (3 min), and 107.4° (5 min).

PF fluid has a higher viscosity (100–170 cPs) than water, so its spreading is slower, resulting in a larger contact angle (Syakur et al., 2012)]. Nevertheless, the corona plasma can still significantly reduce the initial contact angle, especially when treated with a current of 75 mA for 5 minutes.

The hydrophilic properties of bamboo surfaces after plasma treatment are temporary, caused by several factors. Molecular recombination on the material surface, such as polar groups (-OH and -COOH) formed during plasma treatment, can lose their polarity due to interaction with oxygen and environmental humidity (Altgen et al., 2020). In addition, the effectiveness of plasma also depends on the type of modified material and the type of gas (free air) used during treatment (Nascimento et al., 2015)]. The storage environment, including temperature and humidity, also affects the duration of the hydrophilic properties of the bamboo surface (Volpe et al., 1994). A similar phenomenon was reported in European Beech wood, where the contact angle increased from 24.1° to 37.3° two days after plasma treatment (Altgen et al., 2020).

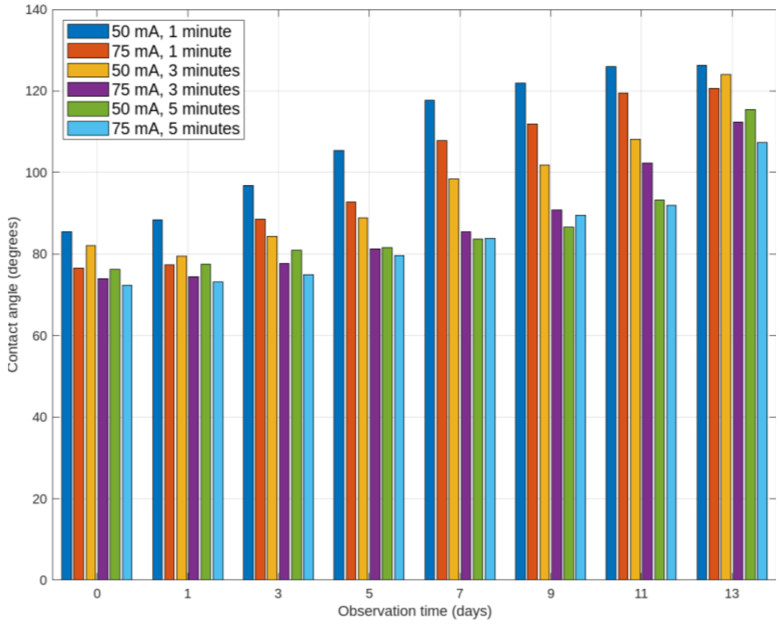


Figure 5. Contact angle of PF after plasma treatments

The statistical analysis showed a significant effect of current strength and plasma corona treatment time on the contact angle value of the bamboo surface. Based on the results of the Independent Sample T Test (**Table 1**), plasma corona treatment with a current strength of 50 mA and 75 mA showed a significant difference (Sig. <0.05). The current strength of 75 mA produced higher surface wettability than 50 mA, which was indicated by a more significant decrease in the contact angle. This is due to the higher current strength, which produces more ions and active radicals to modify the bamboo surface effectively.

Table 1. T Test between 50 mA and 75 mA corona plasma treatments.

Currents	Sig.(2-tailed)	Description
50 mA	0.03	Significance
75 mA	0.04	Significance

The ANOVA results showed that at a current strength of 50 mA (**Table 2**), there was no significant difference between the treatment times of 1 minute, 3 minutes, and 5 minutes. In contrast, at a current strength of 75 mA (**Table 3**), a treatment time of 5 minutes produced a significant difference compared to other treatment times. Corona plasma treatment with a current strength of 75 mA for 5 minutes gave optimal results with the lowest contact angle, reflecting the best wettability on the bamboo surface.

Table 2. ANOVA test of the effect between 50 mA and 75 mA corona plasma treatment time on water fluid.

Corona Plasma discharge	Mean
50 mA, 1 minute	34.9 a
50 mA, 3 minutes	29.7 a
50 mA, 5 minutes	26.1 a
75 mA, 1 minute	28.6 a
75 mA, 3 minutes	24.8 a
75 mA, 5 minutes	19.5 b

Table 3. ANOVA test of the effect between 50 mA and 75 mA corona plasma treatment time on PF fluid.

Corona Plasma discharge	Mean
50 mA, 1 minute	98.3 a
50 mA, 3 minutes	94.9 a
50 mA, 5 minute	87.0 a
75 mA, 1 minute	83.2 a
75 mA, 3 minutes	81.3 a
75 mA, 5 minutes	69.8 b

We can judge that the current strength and treatment time together affect the wettability properties. Higher current strength and longer duration result in more effective surface modification, as indicated by a significant decrease in the contact angle. Corona plasma treatment with a current strength of 75 mA for 5 minutes is the most optimum parameter to improve the wettability of bamboo surfaces, making it ideal for adhesive applications on this material.

3.3 Wettability Properties of Bamboo Surface After Corona Plasma Treatment with 75 mA Current for 5 Minutes

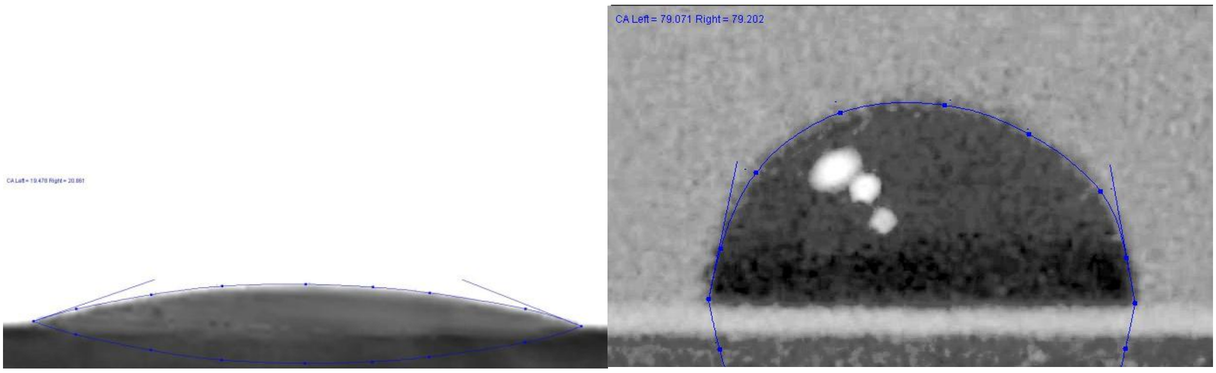
Wettability is the contact angle between a liquid and a material surface, where changes in the contact angle over time indicate the material's ability to absorb liquid (Ahmad et al., 2023). In this study, the wettability of the bamboo surface was observed using water and phenol formaldehyde (PF) fluids after plasma corona treatment with a current strength of 75 mA for 5 minutes. These parameters were chosen because they showed the best results from previous statistical tests. The water wettability image shows that on the 0th day of observation, the initial contact angle of the droplet was 19.9°, below 30°, so the bamboo surface is hydrophilic. On the 13th day of observation, the contact angle increased to 28.5° but was still below 30°, indicating that the surface remains hydrophilic even though its hydrophilic properties tend to decrease over time.

Meanwhile, the PF wettability graph shows that on the 0th day of observation, the initial contact angle was 72.4°, below 90°, so the bamboo surface was partially wet. However, on the 13th day of observation, the contact angle increased to 107.4°, indicating that the surface was hydrophobic again. Water fluid spreads more easily on the bamboo surface than PF fluid because water's viscosity is lower, while the higher viscosity of PF causes slower spreading. The contact angle of PF fluid, which is less than 90°, only lasts until the 9th day (89.6°), and in the following days, the contact angle increases to more than 90°, indicating suboptimal wettability.

The decrease in contact angle over time until reaching saturation value indicates that the wettability properties are influenced by the recombination of molecules on the surface and interaction with the environment (Ahmad et al., 2023). High surface wettability is very important to ensure the ability of PF adhesive to penetrate the bamboo surface and form a strong bond. Contact angles below 90° allow PF adhesives to work more effectively to produce optimal adhesive strength (Wahab et al., 2012)]. Thus, plasma corona treatment can increase the wettability of bamboo surfaces for a certain period, although this property is not permanent.

3.4 Wettability Comparison between Water Fluid and PF Fluid on Bamboo Surface

Applying plasma corona to the bamboo surface affects the fluid contact angle value, where the type of fluid used significantly affects the spreading rate. **Figure 6** shows the difference in contact angle between water and phenol formaldehyde (PF) fluids after plasma corona treatment. Water fluid (a) forms a smaller contact angle than PF fluid (b), indicating that water spreads faster on the bamboo surface. The contact angle of the water fluid is 32°, while the contact angle of PF is 96° to 101°, indicating a significant difference in the wettability properties of the two fluids.



(a) (b)

Figure 6. Surface wettability after plasma radiation at 75 mA, 5 minutes on (a) water, (b) PF

The ability of water to spread quickly is due to its low viscosity and minimal solid content, around 0.03%. The low solid content allows water molecules to move more freely, increasing their ability to spread on the surface (Aksoy et al., 2022). On the other hand, PF fluid has a much higher viscosity and a solid content of up to 45%, which limits the ability of molecules to move freely and slows down the spread of liquid on the surface (Syakur et al., 2012). The high solid content in PF fluid also reduces the wettability of the bamboo surface, which can be seen from its contact angle, which is more significant than 90°.

The reduction in contact angle following plasma treatment is linked to the formation of globular protuberances on the bamboo surface, suggesting surface etching induced by plasma exposure (Wang & Cheng, 2020). Similar effects have been reported in various studies involving plasma-treated materials. For instance, wood subjected to atmospheric-pressure plasma exhibited increased surface roughness (Galmiz et al., 2019), while hot-oil modified wood treated with water vapor plasma also demonstrated enhanced surface properties (Jamali & Evans, 2020). Additionally, polyethylene terephthalate (PET) films modified using radio frequency Ar plasma showed improved hydrophilicity, further supporting the role of plasma treatment in modifying surface characteristics (Xie et al., 2012, as cited in Wang & Cheng, 2020). This increased roughness and the presence of globular protuberances contribute to improved surface water permeability (Prakash et al., 2013).

These findings indicate that while plasma corona treatment enhances the wettability of bamboo surfaces, the type of fluid remains the primary factor governing wettability behavior. Fluids with low solid content, such as water, demonstrate better wettability by forming smaller contact angles, whereas high-solid-content fluids like PF tend to form larger contact angles, reducing surface interaction. This decreased wettability in PF-based fluids poses challenges in industrial applications, particularly in optimizing adhesion performance, where superior wettability is essential for achieving strong bonding between adhesives and substrates.

4. Conclusions

The surface of the bamboo after plasma treatment can increase the surface's wettability. The contact angle value with water fluid on the bamboo surface after corona plasma treatment is less than 30°, so the surface properties of bamboo are hydrophilic. The contact angle value with Phenol-Formaldehyde (PF) fluid on the bamboo surface after corona plasma treatment is less than 90°, so the bamboo surface is partially wet. The application of corona plasma on the bamboo surface is not permanent. The maximum increase in bamboo surface activity is when the bamboo surface is given a corona plasma with a current voltage of 75mA and a treatment time of 5 minutes. The application of corona plasma on the bamboo surface can be done in making composite boards to increase the wettability of the adhesive.

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