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Surface Treatment of Wood Polymer Composites from Straw Fiber Using Argon Plasma Jet Injection: Enhancing Adhesion Properties

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Abstract

The increasing demand for environmentally friendly materials has driven research into biodegradable composites. Straw fiber, an abundant agricultural byproduct, remains underutilized as a filler material in Wood Polymer Composites (WPC). To enhance its application potential, this study investigates the surface treatment of straw fiber/polyvinyl alcohol (PVA) matrix composites using a plasma jet with argon injection. The use of cold plasma as a treatment method is expected to improve the interfacial bonding between the straw fiber and the PVA matrix, thereby enhancing composite properties. The research employs several analytical methods, including morphological analysis of straw fibers before and after plasma jet treatment using Scanning Electron Microscopy (SEM), contact angle measurements, and emission intensity analysis via Optical Emission Spectroscopy (OES). The results reveal significant morphological changes in the straw fiber surface after plasma jet treatment, characterized by increased roughness and improved adhesion with the PVA matrix. The emission intensity analysis identifies peak reactive argon species at $\lambda = 698.223$ nm to $\lambda = 778.398$ nm, N_2^+ at $\lambda = 386.685$ nm to $\lambda = 445.289$ nm, N_2 at $\lambda = 324.768$ nm to $\lambda = 377.983$ nm, and atomic oxygen spectral triplets at $\lambda = 780.341$ nm to $\lambda = 830.867$ nm. Moreover, the decrease in the contact angle from 90° to 57° over 60 minutes demonstrates a transition in the composite's surface properties from hydrophobic to hydrophilic. These findings highlight the effectiveness of plasma jet treatment in modifying straw fiber surfaces for improved composite performance, paving the way for broader applications of biodegradable WPC materials.

INTRODUCTION

Plasma is described as the fourth phase of matter, following solids, liquids, and gases (Lin & Keidar, 2021). Plasma, which contains gases where ions and electrons are separated due to the energy received, is considered reactive (Al-Tayyar et al., 2020; Lin & Keidar, 2021). It has been widely utilized in industries in the form of cold plasma or plasma jets, where electrical energy is delivered between two electrodes at pressures ranging from 10 to 10,000 Pa (Levchenko et al., 2021). Over the last two decades, the application of plasma jets has been increasingly adopted for modifying the surfaces of wood polymer composites (WPCs) (Borrelle et al., 2020). WPC is defined as a material comprising a mixture of polymers and wood fibers (Das et al., 2022). Polymers, which may be natural or

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synthetic, are characterized by high strength and are noted for their rust-resistant and water-resistant properties, making them suitable for structural design, decoration, and wood-polymer construction (Bahrami et al., 2022). In synthetic polymers, ester groups and benzene rings interact with polymer chains, increasing compatibility and molecular distance (Gupta et al., 2022). Cold plasma has been employed to enhance the wettability of polymer surfaces (Iqbal et al., 2021). Numerous attempts have been made to characterize argon jet plasmas under atmospheric pressure, with applied voltage and frequency analyzed to better understand their characteristics. Plasma jets have been generated using capacitively discharged dielectric barriers, with argon gas allowed to flow into ambient air (Macedo et al., 2020).

This cold plasma jet has been applied in various fields, including biology, and has been noted as safe for human use due to its low voltage, making it suitable for food and clinical applications (Silva et al., 2021). To characterize the plasma jet, electron temperatures and compositions have been determined through optical emission spectroscopy. In this study, polyvinyl alcohol, a synthetic plastic with hydrophobic properties and low surface energy, has been added, which does not adhere well to other materials. As a result, surface modification is considered necessary to enhance the wettability of composite materials (Chen et al., 2018). Plasma jets have been applied to composite materials to increase adhesion strength by altering the surface properties of samples exposed to plasma and introducing oxygen-containing polar functional groups (Putra & Susanto, 2021).

Rice straw, classified as a natural fiber consisting of cellulose (40–50%), hemicellulose (20–40%), and lignin, has been proposed as a material for WPC (Alonso-Montemayor et al., 2022). Cellulose serves as the structural backbone of straw, while lignin acts as a protective layer. These components are bound by covalent (anisole) and non-covalent (hydrogen) bonds, forming a lignin-carbohydrate matrix (Al-Tayyar et al., 2020). This lignocellulosic matrix provides wood-like properties (Suhag et al., 2020). WPC is produced by combining wood materials with monomers, which are then polymerized to create materials suitable for construction and automotive applications. Although rice straw has high cellulose and lignin content, which makes it a promising raw material for WPC, its poor compatibility with adhesives has been identified as a significant drawback, leading to reduced mechanical strength (Friedrich, 2022). The mechanical strength of WPC is considered a critical parameter for applications in the automotive and construction industries, and interventions in WPC production methods are deemed necessary to improve compatibility with mixed materials and enhance mechanical performance (Borrelle et al., 2020). Utilizing rice straw in WPC production has been recognized as an effective method for processing and increasing the marketability of this agricultural waste (Maharsih et al., 2021). Although the current methods for producing rice straw-based WPC are limited by inadequate mechanical properties, surface modification using cold plasma technology is proposed as a viable solution to address compatibility issues. This physical modification process is considered simple and scalable, making it feasible for large-scale implementation (Ojeda et al., 2021). Through plasma modification, the surface properties of rice straw are expected to change, improving adhesion to polymers and adhesives during composite production (Harianingsih et al., 2024). Consequently, rice straw-based WPC with excellent mechanical properties and stability could be produced from inexpensive and abundant materials.

MATERIALS AND METHOD

Materials

The rice straw used came from a group of farmers in Central Java, Indonesia, where part of the stem of the "gogo" variety was taken for research. This variety has tough stems and a harvest period of 3-4 months, so its availability is maintained. In this experiment, pure Argon gas with a purity of 99.99% and UHP grade, Polyvinyl alcohol Merck 1.14266.0100, and aquadests were used.

Instrumentation

In this research, jet plasma is utilized in the form of a plasma jet powered by a High Voltage Power Supply (HVPS). The HVPS is connected to an electrode made of a stainless-steel rod with a thickness of 0.15 cm, which is enclosed by a glass tube with an outer diameter of 0.5 cm and an inner diameter of 0.4 cm. At the base, the ground electrode is composed of an aluminium ribbon connected to the ground. The distance between the electrodes is set to 8 cm, with the ground electrode positioned at 0.3 cm from the plasma jet. The voltage supplied ranges between 0–20 kV.Argon gas is flowed through the glass tube at a controlled rate of 3 L/min, while the voltage frequency is maintained at 5.5 kV and 20 kHz. The current-voltage characteristics are measured using a Tektronik TDS 2002 oscilloscope, which is connected to the probe for accurate data collection. The emission intensity was analyzed using Optical Emission Spectroscopy (OES), and the electron temperature is determined through the Boltzmann Eq. (1).

$$ln\frac{I\lambda}{gA} = Const - \frac{Ei}{kT} \quad (1)$$

Where I is the emission intensity, λ is the emission wavelength, g is the statistical weight, A is the transition probability, Ei is the upper level energy of the transition. A is the y axis so that the linear equation y=mx + C is obtained with m being the gradient so that the electron temperature is $1/\mathrm{m}.\lambda ln\frac{I\lambda}{gA}$

The circuit of the plasma jet device can be seen in Figure 1.

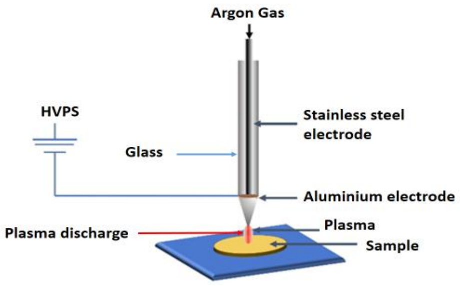


Figure 1. Plasma jet design experiment.

Experiment Procedure

The straw was separated from impurities and washed with clean water. After being washed, the straw was then dried by exposure to sunlight or by using the sun's heat. Once dried, the straw was chopped into smaller pieces and sieved to obtain a size of 1 mm. Straw of uniform size was then exposed to a plasma jet tool. After being exposed to the plasma, the straw was combined with a polyvinyl alcohol matrix. The mixing process was conducted using an internal mixer and compression molding. After mixing until homogeneity was achieved, the mixture was then molded into the desired shape to form WPC. WPC material with dimensions of 60 mm x 20 mm x 0.05 mm was placed in the sample holder, maintaining a distance of 2 mm from the plasma discharge. Contact angle analysis was performed to confirm the change in surface properties to hydrophilic by applying a single water droplet to the WPC surface, both before and after plasma treatment. The surface roughness of the polymer materials was measured using a LEO (500)/Zeiss field-emission scanning electron microscope (SEM), while emission intensity was analyzed using Optical Emission Spectroscopy (OES).

RESULTS AND DISCUSSION

Plasma Emission Intensity

The plasma jet method with argon injection, as demonstrated in this research, has been shown to effectively modify the surface of straw fibers by increasing surface roughness, incorporating polar functional groups, and transitioning the surface from hydrophobic to hydrophilic (Miebach et al., 2022). Compared to alternative methods, several distinctions can be observed. Chemical treatment is often employed to modify straw fibers through immersion in chemical solutions such as NaOH, which removes lignin and hemicellulose (Mohammadi et al., 2024). However, this method produces chemical waste and carries a risk of fiber degradation. UV-ozone treatment is another option, in which surfaces are exposed to UV light in the presence of ozone, leading to increased hydrophilicity (Nadeem et al., 2024). While this method is environmentally friendly, it primarily modifies the surface chemistry without significantly altering topography. Laser ablation has also been used to achieve high surface roughness through precision microstructuring (Salajková et al., 2021). Although it produces highly controlled physical modifications, it lacks chemical functionalization and requires expensive equipment. Thermal treatment, involving the application of heat, has been used to restructure fibers thermally, improving their adhesion properties, but it poses the risk of thermal degradation (Tang et al., 2024). Among these methods, the plasma jet has been shown to provide a unique combination of chemical and physical modifications without producing chemical waste (Miebach et al., 2022). While the equipment required for plasma treatment is specialized, the method is more versatile and environmentally friendly compared to chemical treatment or laser

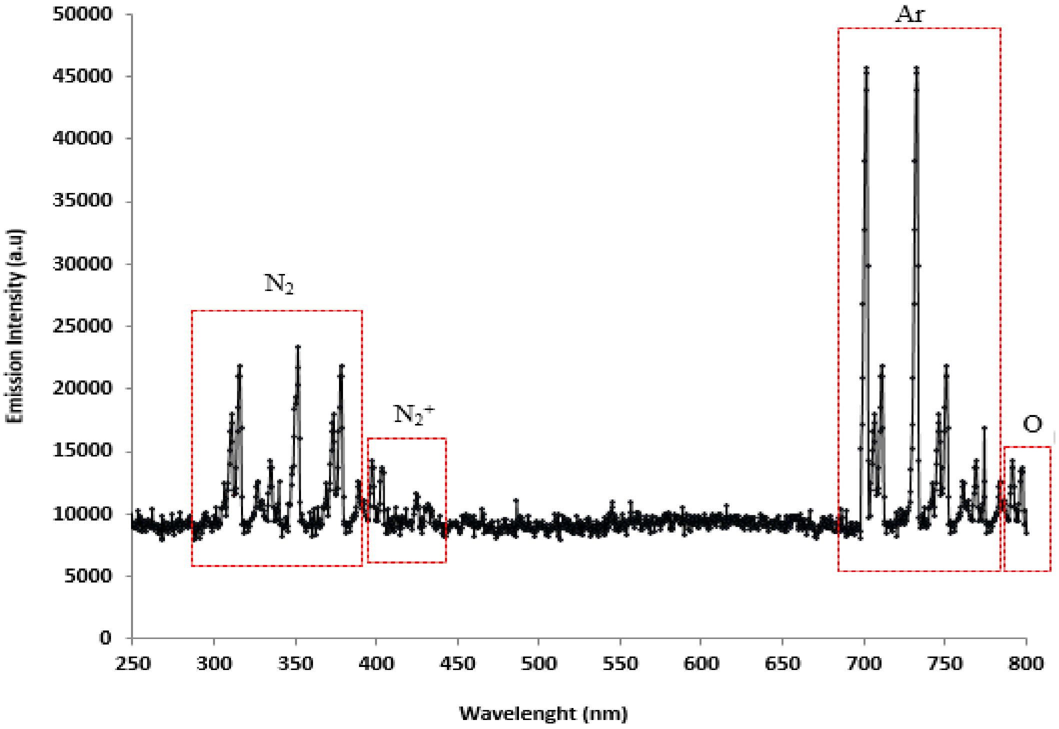
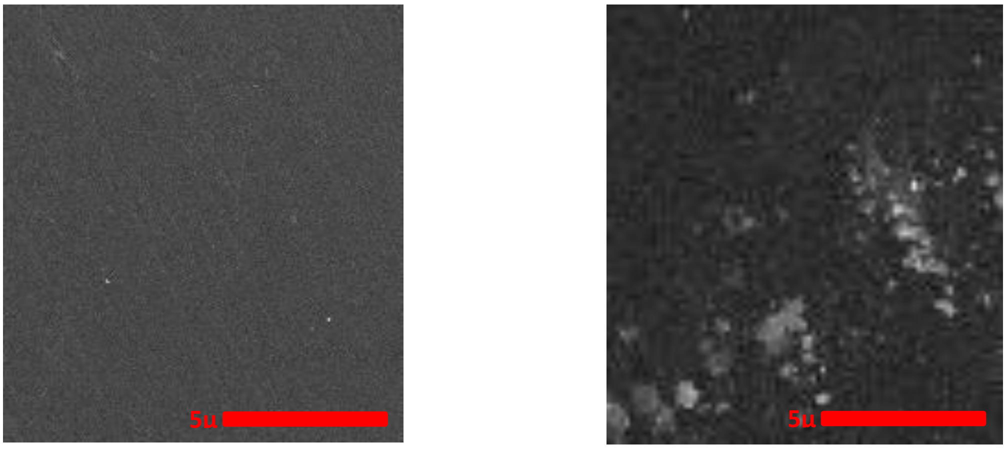


Figure 2. Optical emission spectra of jet plasma with argon injection.

ablation. The comparison highlights the potential of plasma jet treatment as an optimal method for enhancing the surface properties of straw fibers in wood polymer composites. In this research, the plasma jet only uses pure argon, but the plasma chemistry is a mixture of argon–nitrogen–oxygen. This means that plasma release in open air is always influenced by the chemistry of the surrounding air (Wang & Sun, 2016). Based on the ionization rate for an atmospheric plasma discharge, most of the plasma energy is spent to produce optically permitted states, forbidden (metastable) states, and dissociation. Most metastables have long life spans, so their populations can increase to high levels.

As shown in Figure 2, the spectral data from this study indicate that argon plasma is dominated by strong emission from excited neutral argon atoms, primarily through depopulation of the 4p energy level via several 4p–4s transitions, with the most prominent transitions occurring between λ = 698.223 nm and λ = 778.398 nm. This observation aligns with the findings of Roomy & Murbat (2023), who also reported dominant emissions from neutral argon atoms in plasma jets, with the emission intensity primarily dependent on metastable argon states. Unlike atomic argon ions, which exhibit negligible emission in this study, weak ion emissions under higher ionization conditions, suggesting that the ionization energy threshold for argon in open-air plasma jets is not frequently achieved (Roomy & Murbat, 2023). For nitrogen species, the dominant emissions were detected from molecular nitrogen ions (N_2^+), specifically within the first negative system, spanning wavelengths from λ = 386.685 nm to λ = 445.289 nm. This result is consistent with observations by Wang and Sun (2016), who reported strong N_2^+ emissions in dielectric barrier discharge plasmas, highlighting the significant role of metastable collisions and neutral direct excitations in producing these emissions. The secondary dominant emissions, attributed to nitrogen molecules (N_2), were detected between λ = 324.768 nm and λ = 377.983 nm, further confirming the role of low-energy electrons in nitrogen excitation processes. Regarding oxygen species, this study recorded weak atomic oxygen emissions, characterized by spectral triplets between λ = 780.341 nm and λ = 830.867 nm, while molecular oxygen emissions were even weaker. This aligns with findings by Lu et al. (2023), who observed minimal oxygen molecular emissions in argon-dominated plasma environments, attributing this to the lower excitation cross-section of molecular oxygen compared to nitrogen or argon species (Lu et al., 2023).

In comparison to other methods, such as dielectric barrier discharge (DBD) or microwave plasma, jet plasma demonstrates a unique spectral behavior due to the open-air influence, as



(a) (b)

Figure 3. SEM images of untreated (a) and treated plasma (b).

surrounding atmospheric gases contribute significantly to plasma chemistry. Studies such as those by Lu et al. (2023) have shown that DBD plasmas in controlled environments yield more uniform ionization and stronger molecular oxygen emissions due to the absence of atmospheric nitrogen interference. This further highlights the versatility of jet plasma in achieving specific reactions under ambient conditions, although it may lack the precision of controlled systems

Surface Morphology

SEM analysis was carried out to observe the surface morphology of WPC-PE before and after plasma jet treatment with argon injection. SEM analysis can observe changes in surface topography and reduction of surface roughness during the argon injection process. SEM results of the WPC-PE surface before and after plasma treatment can be seen in the Figure 3.

The surface texture of Wood Polymer Composite (WPC) is shown to undergo significant changes after plasma modification. Initially, the WPC surface is observed to be smooth, but it becomes rougher after plasma treatment due to the etching process. This phenomenonis caused by plasma-induced etching, which results in contour changes and the formation of graded structures (Harianingsih et al., 2024). Heat is generated when injected plasma collides with the WPC surface, leading to the degradation of polymer bonds, particularly on lignin surfaces. As a result, a peeled or graded texture is produced, which enhances surface roughness. An increase in surface roughness in plasma-treated polymer composites, where strong interactions occur between reactive plasma species and electrons with the surface (Yáñez-Pacios & Martín-Martínez, 2017). This interaction is found to facilitate stronger bonding between the polyvinyl alcohol (PVA) matrix and the WPC surface, making the composite more cohesive and durable. Furthermore, plasma interaction with wood components has been observed to reduce surface roughness in certain areas by selectively ablating lignin, exposing more polymer. Surface abrasion caused by plasma jet treatment leads to changes in surface topography, with the degree of roughness influenced by operational parameters such as platform speed (Zhou et al., 2022). Lower platform speeds are shown to produce smoother surfaces due to reduced plasma exposure. Additionally, functional groups are introduced to the surface through plasma treatment, as demonstrated by Gupta et al. (2019). In this process, polymer surfaces are irradiated with positive ions and reactive species, enhancing their reactivity and adhesion properties. These improvements are considered essential for applications requiring robust composite materials, as strong interfacial bonding between the polymer matrix and the filler is ensured. When compared to other surface modification methods, such as chemical treatment or mechanical abrasion, plasma treatment is recognized for its precise control over surface roughness and chemical reactivity. Unlike chemical methods, which might achieve similar adhesion properties, the plasma jet's ability to create graded structures through controlled etching is considered a unique advantage, particularly for applications requiring tailored surface topography.

Contact Angle

The relationship between contact angle and treatment time was analyzed, and it was

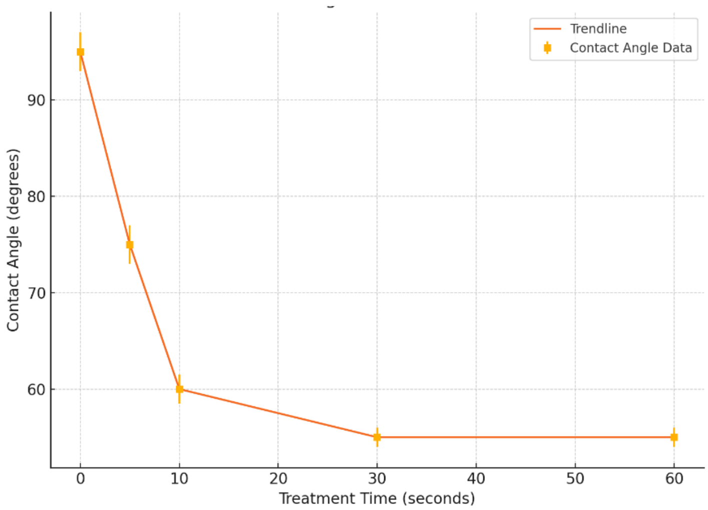


Figure 4. Correlation between contact angle with treatment time.

observed that the contact angle significantly decreased during plasma treatment. As shown in Figure 3, the contact angle of the surface was initially measured at approximately 95° before plasma exposure. However, within the first 10 seconds of treatment, the contact angle drastically dropped to approximately 60°. This rapid decline indicates that the surface underwent substantial modification, becoming more hydrophilic due to the plasma treatment. The decrease was attributed to the introduction of polar functional groups, such as -OH and -COOH, caused by interactions between reactive plasma species and the surface material (Miebach et al., 2022). After 10 seconds, the contact angle stabilized at approximately 55°–60° despite continued plasma exposure for up to 60 seconds. This suggests that the surface reached a saturation point in its modification, and further treatment did not result in additional changes. The stabilization can be explained by the complete coverage of the surface with reactive groups, rendering it maximally hydrophilic. This phenomenon occurs because plasma treatment involves the bombardment of the surface with reactive species, including ions, electrons, and radicals. These species interact with the surface material, breaking chemical bonds and introducing new functional groups that enhance the surface's polarity. During the initial phase of treatment, the plasma energy is primarily used to initiate these chemical changes. As treatment progresses, a saturation effect is observed, as the surface becomes fully functionalized and no additional reactive sites are available. Furthermore, the high energy of the plasma can remove contaminants or smooth out micro-level irregularities, contributing to the overall change in wettability. These findings demonstrate that plasma treatment is an effective technique for surface modification, achieving significant results in a short duration while avoiding overprocessing.

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In Figure 4, it is shown that the surface characteristics of WPC are changed from hydrophobic to hydrophilic after 60 seconds of plasma treatment. This change is indicated by a decrease in the contact angle from 95° to 57°. A very significant decrease in the contact angle is observed within the first 12 seconds, after which it remains relatively stable despite the continued increase in plasma treatment time. The hydrophobic nature of the WPC surface is altered to hydrophilic due to the bombardment of positive ions, electrons, and reactive species generated by the plasma jet discharge. This process also results in increased surface adhesion. However, the penetration of water vapor is not completely prevented, as this technique is unable to effectively coat the wood capillaries, leaving the WPC surface partially susceptible to water absorption. In addition, the measurements of the contact angle demonstrate that plasma discharge modifies the morphology of the wood, increases the thickness of the surface film, and roughens the texture. The hydrophilic nature of the WPC surface enhances interaction with liquids, leading to improved adhesion to layers of paint or other adhesive materials. This results in better color stability for the WPC material. The reduction in the contact angle of water droplets, which decreases from approximately 80° to 40° after plasma treatment, indicates an increase in hydrophilicity and adhesion properties. These improvements are attributed to changes in the surface chemistry, energy, and topography. Furthermore, the exposure of the WPC surface to plasma discharge makes it more reactive. The interaction between the reactive surface and the atmosphere leads to the inclusion of oxygen-containing reactive species on the WPC surface. This process results in the formation of polar functional groups, including C=O, OH, O=C=O, COOH, peroxide, and others (Berthomieu & Hienerwadel, 2009). These polar functional groups strengthen and broaden the bonding capabilities of the WPC surface. Additionally, when the contact angle is reduced to $\theta \leq 90°$, the degree of imbibition is increased, resulting in enhanced energy.

CONCLUSION

Plasma treatment has been demonstrated as an effective method for modifying the surface properties of Wood Polymer Composite (WPC) from rice straw fiber, transitioning it from hydrophobic to hydrophilic. The reduction in contact angle, as observed in the study, highlights the introduction of polar functional groups such as -OH, C=O, and COOH, which significantly enhance the surface energy, adhesion, and reactivity of the WPC surface. These modifications are achieved through the bombardment of positive ions, electrons, and reactive species during plasma discharge. While the treatment effectively improves hydrophilicity and adhesion. Furthermore, plasma treatment increases surface roughness and alters morphology, thereby facilitating stronger bonding with paints, adhesives, and other surface coatings. Compared to traditional methods, plasma treatment offers unique advantages by simultaneously enhancing the chemical, energy, and topographical properties of WPC surfaces, making it a versatile and environmentally friendly technique for surface modification.

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