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The Effect of Silane Treatment on Interfacial Shear Strength and Wettability of Luffa Cylindrica Fiber

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Abstract

Luffa cylindrica, a plant from the Cucurbitaceae family, is abundant but remains underutilized. Its simple fiber extraction and high cellulose content make it a potential eco-friendly reinforcement material for composites. This study examines the effect of silane concentration on the interfacial shear strength (IFSS) and wettability of Luffa cylindrica fibers. The fibers were treated with silane solutions at concentrations of 0.5%, 1%, and 1.5%. Tests included IFSS, wettability, and SEM analysis. Results show that silane treatment improves both IFSS and wettability, with the highest values obtained at 0.5% concentration (3.68 MPa and 43.94 mN/m, respectively). SEM observations confirmed better fiber–matrix adhesion at this concentration, where multiple matrices were attached and embedded on the fiber surface. These findings highlight the potential of silane-treated Luffa cylindrica fibers as effective reinforcements for composites.

1 Introduction

In recent decades, the development of composite technology using natural fibers as reinforcement has grown rapidly. This progress is primarily driven by an increasing awareness of the environmental impact caused by synthetic fibers, leading researchers and industries to consider natural fibers as a more eco-friendly alternative [1]. Luffa cylindrica, a plant belonging to the cucumber family, is widely distributed in tropical regions. It is cultivated for both fiber production and food consumption[2]. The fibers of this plant consist of approximately 60% cellulose, 30% hemicellulose, and 10% lignin[3]. These components make it a promising material for composite reinforcement. However, natural fibers also have inherent limitations. They typically exhibit weak bonding with the matrix and high water absorption. These characteristics reduce the mechanical properties of the composites, resulting in poor fiber–matrix adhesion [4]. To overcome these drawbacks, natural fibers are often subjected to physical or chemical treatments that alter their structure and surface properties. Such treatments aim to enhance mechanical strength and improve interfacial bonding. For example, heat treatment increases the fiber crystallinity index and strength, while alkali treatment modifies the fiber structure to achieve improved mechanical performance [5]. Another important factor that influences composite properties is the interfacial shear strength between the fiber and the resin. This property is affected by the type of fiber and matrix, as well as the surface treatment applied to the fiber[6]. Excessive water content in the fiber reduces the strength of fiber–matrix bonding[7]. Chemical treatment is therefore considered essential, as it improves fiber properties by modifying their microstructure[8]. Untreated natural fibers, due to their hydrophilic nature, show poor compatibility with matrices. Physical and chemical modifications can significantly enhance adhesion, fiber strength, and the overall mechanical properties of composites [9].

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Among the various chemical treatments, silane treatment is recognized as one of the most effective methods. It improves fiber strength, enhances surface structure, and strengthens the fiber–matrix bonding [10]. Silane molecules contain reactive functional groups that interact with the surface of natural fibers to form chemical bonds, while also bonding with the matrix. This dual function makes silane a strong connector between fiber and matrix[11], [12]. Common reactive groups in silane include hydrogen atoms (hybrid silicon), chlorine atoms (dichlorosilane), hydroxyl groups (silanol), and alkoxy groups such as methoxy- or ethoxy-silane[13].



Figure 1. a) Luffa cylindrica b) Luffa cylindrica fiber

Silane treatment not only enhances the physical and mechanical properties of natural fibers but also makes them more effective as reinforcement in composites. Luffa cylindrica fibers are particularly interesting due to their high cellulose content and the relatively limited amount of research conducted on them. This study, therefore, focuses on evaluating the interfacial shear strength (IFSS) and wettability of silane-treated Luffa cylindrica fibers.

2 Research Methods

2.1 Materials

Luffa cylindrica fiber was used as the main material for analyzing mechanical properties. The fibers were sourced from local farmers in Seputih Village, Mayang Subdistrict, Jember Regency.

2.2 Silane Treatment

Luffa cylindrica fibers were treated with silane by soaking them in solutions with concentrations of 0.5%, 1%, and 1.5% at room temperature for 4 hours. After soaking, the fibers were rinsed with distilled water until a neutral pH (7) was reached. The rinsed fibers were then air-dried at room temperature for 48 hours and subsequently oven-dried at 70 °C for 10 hours. The silane chemical treatment method used in this study refers to the methodology applied by Raharjo et al [14]

2.3 Testing Interfacial Shear Strength (IFSS)

The interfacial shear strength (IFSS) of Luffa cylindrica fibers was evaluated using a shear stress test. Samples were prepared by placing the fibers between two sheets of HDPE film on an aluminum mold. The samples were then heated at 150 °C for 15 minutes under a pressure of 1.5 bar using a hot press. To embed the fibers into the matrix, a hole was drilled so that 1–2 mm of fiber length was positioned inside (Figure 2). Before testing, the samples were mounted on cardboard. IFSS tests were conducted using a JTM UTS510 universal testing machine equipped with a 10 kg load cell and a crosshead speed of 1 mm/min. The IFSS test refers to the method applied by Lee et al [15]. For each variation, 30 specimens were tested. The IFSS value was calculated using the following equation:

$$\tau_{app} = \frac{F_{max}}{\pi D L} \quad (1)$$

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τ_{app} = Shear strength of fiber and matrix interface (MPa), F_{max} = load (N), D = Fiber Diameter (mm), dan L = Fiber embedded into the matrix (mm)



Figure 2. Specimen Test IFSS

2.4 Wettability

Fiber surface properties were examined using an Olympus Stereo Microscope (type SZ 1145 TR). The droplet volume was controlled at 1.5 ml using micropipettes. Distilled water and ethylene glycol were applied as test liquids to investigate the fiber surface. The wettability analysis was conducted using five replicates for each treatment group. For every replicate, contact angle measurements were taken at four different locations on the fiber surface. The resulting data were then used to calculate the surface energy in accordance with the procedure outlined by Chen et al [16]. Fiber surface energy was estimated from contact angle values (θ) using the Owens–Wendt method, as shown in Equation (2) [17]:

$$y_{L(1+\cos\theta)} = 2\sqrt{y_S^d}\sqrt{y_L^d} + 2\sqrt{y_S^p}\sqrt{y_L^p} \quad (2)$$

In this equation, the superscripts d and p represent the dispersive and polar components of surface energy, respectively, while the subscripts S and L denote solid and liquid states. For each variation, five specimens were tested.

2.5 Scanning Electron Microscopy (SEM)

The surface morphology of fibers embedded in the matrix after IFSS testing was examined using a scanning electron microscope (SEM, Jeol JCM-7000). The samples in this test were taken from one sample in each variation after the IFSS test was conducted. Prior to observation, the samples were coated with a thin layer of platinum to improve surface conductivity and imaging quality.

3 Result and Discussion

3.1 Wettability

Wettability testing was conducted to investigate the impact of silane treatment on the surface energy of Luffa cylindrica fibers. As illustrated in **Figure 3**, silane-treated fibers (SL) were used for contact angle measurements. The tests were performed using probe liquids and observed under a stereo microscope, resulting in macroscopic images of the fiber surface. The contact angle was determined by analyzing the shape of liquid droplets attached to the fiber. A smaller contact angle indicated that the liquid spread more easily across the surface. Thinner or flatter droplets suggested a stronger interaction between the probe liquid and the fiber, reflecting improved wettability after silane treatment.

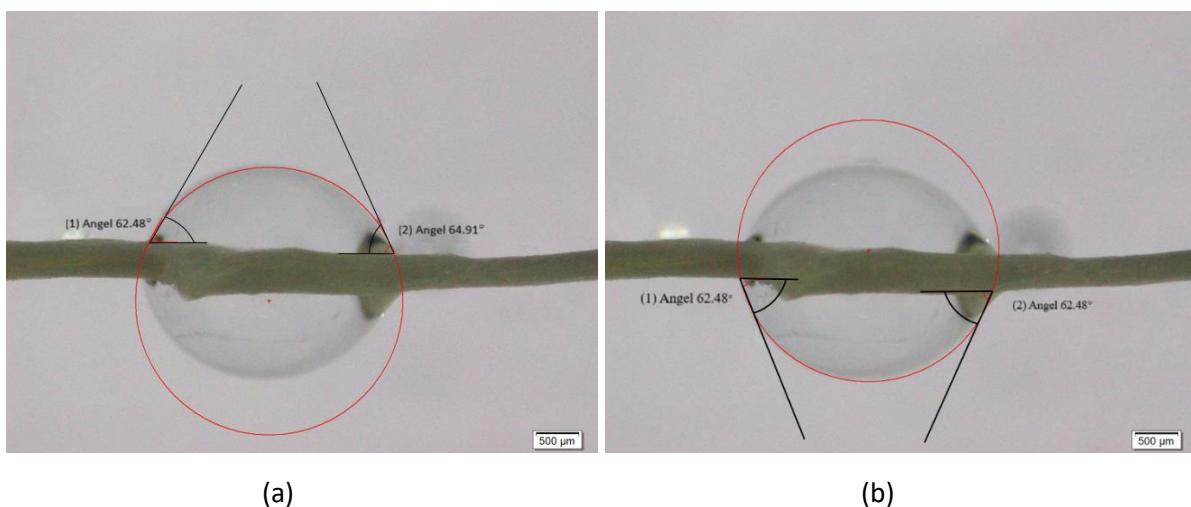


Figure 3. The result of Macro droplet Investigator Liquid observation: a) Upper View; b) Bottom View

Table 1 shows the contact angle results obtained from the tests and the corresponding surface energy calculations. The contact angle decreased as the silane solution concentration was reduced. The highest contact angles were recorded in untreated fibers (TP), with values of 71.59° in distilled water and 70.32° in ethylene glycol. These high values indicate poor wettability, as the liquids had difficulty spreading across the fiber surface due to their low polarity. The presence of non-polar components such as wax, oil, and lignin on the fiber surface also contributed to this weak interaction.

In contrast, the lowest contact angles were observed in fibers treated with a 0.5% silane solution (SL05), which exhibited contact angles of 66.14° in distilled water and 64.09° in ethylene glycol. This improvement can be attributed to the reaction between the polar functional groups of silane and the hydroxyl groups of the *Luffa cylindrica* fibers, which enhanced the compatibility of the fiber surface with polar liquids. As a result, liquids were able to spread more easily, indicating better wettability.

Overall, silane treatment was effective in reducing contact angles and improving fiber wettability by modifying the surface characteristics of the fibers. However, as silane concentration increased beyond 0.5%, the contact angle values also increased. This suggests that excessive silane may form a dense coating layer, reducing the availability of reactive sites and thus limiting liquid penetration. These findings are consistent with the results reported by Raharjo [14], confirming that an optimal concentration of silane treatment enhances wettability, whereas higher concentrations may have a diminishing effect.

Table 1. Contact Angle Measurement Results and Calculation of *Luffa Cylindrica* Fiber Surface Energy.

Specimen	Contact angle (θ)		Dispersion (mN/m)	Polarity (mN/m)	Surface Energy (mN/m)
	Distilled water	Ethylene glycol			
TP	71.59	70.32	2.08	37.10	39.18
SL05	66.14	64.09	2.68	41.25	43.94
SL1	68.39	67.69	1.96	41.15	43.11
SL15	71.18	69.88	2.11	37.45	39.57

Table 1 presents the surface energy values of untreated *Luffa cylindrica* fibers (TP) and silane-treated fibers (SL). The highest surface energy was observed in fibers treated with 0.5% silane solution (SL05), reaching 43.94 mN/m . This increase reflects the improved interaction between the fiber surface and polar liquids, indicating enhanced wettability after silane modification.

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However, as the silane concentration increased to 1% (SL1) and 1.5% (SL15), the surface energy values decreased. This trend suggests that excessive silane may form a thicker coating layer on the fiber surface, reducing the exposure of reactive functional groups. As a result, liquid penetration becomes limited, and the effectiveness of surface modification decreases.

These findings confirm that silane treatment significantly improves the surface properties of Luffa cylindrica fibers. Nevertheless, the results also highlight that optimal performance is achieved at lower silane concentrations, particularly 0.5%. Beyond this level, the benefits of treatment decline due to over-saturation of the fiber surface.

3.2 Interfacial Shear Strength (IFSS)

The results of IFSS testing are presented in **Figure 4**, which shows the relationship between silane treatment of Luffa cylindrica fibers and their interfacial shear strength (IFSS). This test was performed to evaluate the bonding quality between the fibers and the matrix. As shown in **Figure 4**, silane-treated fibers (SL) exhibited higher IFSS values than untreated fibers (TP). The interfacial shear strength values obtained were 2.91 MPa for TP, 3.68 MPa for SL05, 3.30 MPa for SL1, and 3.01 MPa for SL15. These results indicate that silane treatment improved the interfacial shear strength of Luffa cylindrica fibers by 3.08%–26.03%. The highest strength was achieved with 0.5% silane treatment (SL05), while the lowest was observed in untreated fibers (TP).

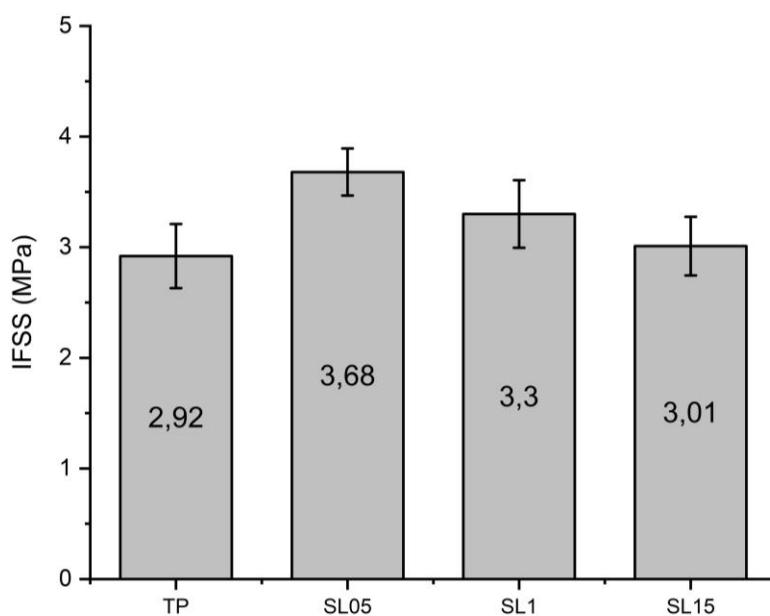


Figure 4. Interfacial Shear Strength of Luffa Cylindrica Fiber

The increase in shear strength can be attributed to the addition of silane, which coated and filled the pores on the fiber surface, forming a stronger bond with the matrix[18]. This treatment made the fiber surface denser and more uniform, thereby enhancing fiber–matrix adhesion compared to untreated fibers. This phenomenon was also supported by SEM observations (**Figure 5b**), which showed large amounts of matrix attached to the fiber surface after silane treatment.

However, the IFSS values decreased when the silane concentration was increased beyond 0.5%. Excess silane led to aggregation on the fiber surface, increasing fiber diameter and creating a rough, uneven texture. These changes reduced wettability and weakened the interfacial bonding between the fiber and the matrix[14]. Overall, the IFSS values were consistent with the surface energy results presented earlier (**Table 1**). As surface energy increased, fiber–matrix bonding improved, demonstrating a direct correlation between wettability and interfacial shear strength.

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3.3 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) was performed to observe the surface topography of *Luffa cylindrica* fibers after interfacial shear strength (IFSS) testing. The SEM images of untreated and silane-treated fibers are presented in **Figure 5**, showing the fiber–matrix interface after testing. These images were taken from fibers embedded in the matrix to highlight the quality of the interfacial bond.

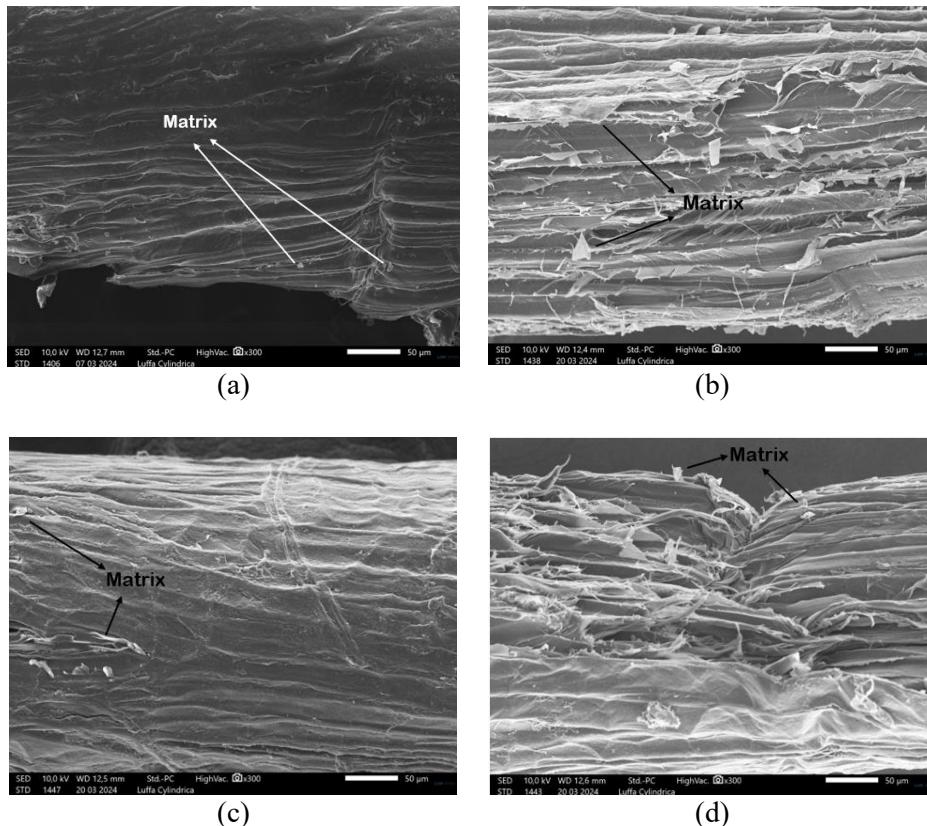


Figure 5. SEM Observation Results of *Luffa Cylindrica* Fiber Surface After IFSS Testing a) Without Treatment b) Silane Treatment 0,5% c) Silane Treatment 1% d) Silane Treatment 1,5%

In **Figure 5a**, untreated fibers show only a small amount of matrix attached to the fiber surface, indicating weak interfacial bonding. In contrast, **Figure 5b** (SL05) demonstrates a significant improvement, with a larger amount of matrix adhering to the fiber surface. This confirms the formation of a strong interfacial bond at 0.5% silane concentration. However, **Figures 5c** (SL1) and **5d** (SL15) show a reduction in matrix adhesion compared to SL05, suggesting that higher silane concentrations decrease bonding effectiveness. The improvement observed at 0.5% silane concentration is attributed to the formation of a uniform silane coating on the fiber surface. The silane layer effectively fills the grooves of the fiber, resulting in a smoother and more solid interface [17]. At higher concentrations, however, excessive silane accumulates on the fiber surface, increasing fiber diameter and creating an uneven texture [14]. These SEM observations support the IFSS results shown in **Figure 4**, where greater matrix adhesion corresponded to stronger interfacial bonding between the fiber and the matrix.

4 Conclusion

This study demonstrates that silane treatment significantly enhances the surface properties and interfacial bonding of *Luffa cylindrica* fibers. A concentration of 0.5% was identified as the optimum, yielding the highest interfacial shear strength (IFSS) of 3.68 MPa and surface energy (indicating wettability) of 43.94 mN/m. This enhancement is attributed to the formation of a uniform silane layer on the fiber surface, which improves fiber-matrix adhesion and compatibility with polar resin systems. The

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quantitative results from IFSS and wettability tests are strongly supported by SEM analysis, which visually confirmed superior interfacial bonding at the 0.5% concentration, evidenced by a homogeneous silane coating and extensive matrix residue adhering to the fiber surfaces.

Conversely, silane concentrations exceeding 0.5% led to a decline in performance. The higher concentrations (1% and 1.5%) resulted in silane agglomeration on the fiber surface, increasing surface roughness and fiber diameter, while reducing the availability of reactive sites. This oversaturation consequently diminished both wettability and IFSS.

In conclusion, these findings confirm that an optimal silane concentration is critical for maximizing performance. The 0.5% silane treatment provides the most effective improvement in the adhesion and mechanical interaction at the fiber-matrix interface. This research highlights the potential of optimally treated *Luffa cylindrica* fibers as an eco-friendly reinforcement material, providing a crucial foundation for developing sustainable composite materials.

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