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EFFECT OF SILANE COUPLING AGENT TREATMENT FOR PALM EMPTY FRUIT BUNCH AND BAMBOO FIBER

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Abstract:

Fiberglass is a synthetic material commonly used in construction, mainly for building and heating, ventilating, and air-conditioning (HVAC) systems. However, its use poses potential health and environmental risks. Bio-based materials such as palm empty fruit bunch (PEFB) and bamboo fiber (BF) are promising natural insulation materials, providing a sustainable substitute to the frequently used fiberglass in HVAC ducting. The aim of this study was to characterize the physical and chemical properties of nonwoven bamboo fiber (BF) and Palm Empty Fruit Bunch (PEFB), both untreated and treated, as insulation materials for interior applications. The untreated BF and PEFB fibers, and the sample fibers treated with silane coupling agent, were subjected to physical characterization in terms of hydrophobicity using water contact angle (WCA), morphology using scanning electron microscopy (SEM), thermal decomposition using thermo-gravimetric Analysis (TGA), chemical functional groups using Fourier transform infrared spectroscopy (FTIR), and thermal conductivity. The results suggest that the treated PEFB and BF display higher water repellence, improved thermal stability, and acceptable thermal conductivity. Therefore, treated PEFB and BF using silane coupling agents have a high potential to sustainably substitute the conventional FG as insulator for HVAC systems.

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Keywords:

Palm Empty Fruit Bunch (PEFB); Silane Coupling Agents; Thermal Conductivity; Composite Fiber; Hydrophobic; Thermal Decomposition; Bio-Based Insulation

Introduction

Heating, ventilation, and air-conditioning (HVAC) is a critical part of the architecture, engineering, and construction (AEC) sector in delivering thermal comfort and maintaining indoor air quality (IAQ) to conditioned space. However, persistent challenges associated with fire resistance and thermal efficiency of the insulation materials used in HVAC systems have intensified the need for a comprehensive investigation and innovative solutions. The issue of fire resistance in HVAC insulation materials is of paramount concern, as conventional materials may not meet the stringent safety requirements demanded by modern building codes and regulations. Instances of HVAC-related fires have raised serious questions about the efficacy of existing insulation materials, emphasizing the critical need for advancements in this domain. Current fire regulations and building standards must be updated to cater to the evolving nature of fire risks in HVAC systems.

Simultaneously, the thermal efficiency of HVAC systems remains a pressing challenge, with implications far beyond immediate safety concerns. Conventional insulation materials may not achieve design thermal conductivity during operation, leading to an increase in energy consumption, utility costs, and carbon emissions. The imperative to mitigate climate change and promote sustainable practices necessitates a re-evaluation of insulation materials to align with energy-efficient HVAC solutions. In the quest for innovative solutions, a significant gap exists in the exploration of insulation materials that seamlessly blend fire resistance and superior thermal performance. The conventional materials, while individually addressing either fire resistance or thermal efficiency, often compromise one aspect for the other. Bridging this gap requires a holistic approach that not only ensures the safety of occupants but also contributes to the overarching goals of energy conservation and environmental sustainability.

To tackle these challenges, it is important to understand the basics of HVAC systems and scrutinize the materials that form their backbone. The complexity of the piping and the ducting layout necessitates the use of insulation materials with sufficient fire resistance and efficient thermal transfer, which requires a deep understanding of thermodynamics and fire engineering. The challenges associated with fire resistance and thermal efficiency in HVAC systems underscore the need for innovative solutions. The integration of novel materials into HVAC systems presents a promising avenue for addressing these challenges. The materials under consideration are the Palm Empty Fruit Bunches - Fiberglass (PEFB-FG) composite, and the Bamboo Fiber (BF). Therefore, the research aims to evaluate the performance of both materials as a sustainable, bio-based alternative to conventional insulation materials.

A few studies were undertaken to evaluate the effectiveness of composites with natural fiber to absorb sound in many applications. Natural fibers, namely wood, sisal, flax, hemp, and bamboo (Karthi et al., 2020), are currently considered significant plant fibers with significant potential in polymer composite materials for sound absorption (Prakash, 2020). Bamboo fiber is a versatile composite material due to its mechanical toughness, thermal stability, good sound absorber, and ease of chemical modification (Bhingare & Prakash, 2022). It has been

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demonstrated that rigid PU composite bamboo fibers are effective sound absorbers at low frequencies in the range of 100- 2000 Hz of sound absorption coefficient (Baek & Kim, 2020). RPU is also used as an acoustic material due to its porosity as effective sound absorption at low and broad frequency ranges (Chanlert & Ruamcharoen, 2021), as well as being capable of dissipating sound energy at the frequency range (Sukhawipat et al., 2022)

Moreover, bamboo fiber has a low number of surface hydroxyl groups and a large particle size, which restricts its involvement in the polyurethane foaming reaction (Qiu et al., 2021), and its hydrophilic nature may limit its applications (Qui et al., 2021). Chemical treatments on bamboo fiber to reduce its hydrophilic nature include alkalization (Sugiman, Setyawan, & Anshari, 2019), peroxide (Abd Halip et al., 2019), and silane (Siy et al., 2023). The fiber is improved from hydrophilic to superhydrophobic, achieving water contact angle of more than 150°. For lightweight roof applications, the RPU composite doped with untreated and treated bamboo fiber was found to be able to reduce moisture absorption at the surface structure and increase sound absorption (Sharma et al., 2023) The rigid PU composite is also seen as a potential alternative to fiberglass in sound absorption for roof insulation (Roseli et al., 2024).

This study aims to shed light on the potential of PEFB and bamboo fiber composites as sustainable bio-based candidate materials for HVAC by conducting physical and chemical comparative analyses between the two novel materials. The exploration of composite insulation materials offers a promising avenue to bridge the existing gaps and redefine the benchmarks for safety and efficiency in HVAC systems, to foster safer, more energy-efficient, and environmentally conscious HVAC systems.

Methodology

Materials

The raw material used in this study was *Gigantochloa Scortechinii* bamboo fiber (BF) species obtained from a private company making products from bamboo materials located in north peninsular Malaysia. Palm empty fruit bunch (PEFB) fiber was sourced from a factory situated in the southern part of peninsular Malaysia. The sample of fiber was prepared with a diameter of approximately 0.3 mm, extracted from culm strips, and cut to a length of 100 mm. Meanwhile, waste PEFB fiber has a diameter of approximately 0.4 mm, with an average length of about 80 mm.

Sample Preparation

Both nonwoven untreated and treated BF and PEFB fibers were modified using a silane coupling agent to resist water absorption on the surface fiber (Roseli et al., 2023). The chemical treatment of both fibers started with preparing a solution consisting of ethanol and distilled water, in 4:1 weight ratio, and then was blended with 7 drops of 3-aminopropyl (diethoxy) methylsilane hydrolyzed. This solution was stirred continuously until it achieved homogeneity. Subsequently, 10 g of fibers were incorporated into the solution and stirred continuously (silane-filler ratio of 1: 100). The fibers were immersed in silane solution for 3 hours, after which the pH was adjusted to 4 using the modified acetic acid method [10]. The BF was then oven-dried at 80°C for 24 hours. Fig. 1 depicts a schematic illustration of the silane treatment procedure for BF, which was also applied to PEFB fiber. The samples were categorized as untreated PEFB (UPEFB), treated PEFB (TPEFB), untreated bamboo fiber (UBF), and treated bamboo fiber (TBF).

Physical Characterization

Hydrophobic Measurement

The hydrophobic fiber property was determined by water contact angles of both nonwoven untreated and treated UPEFB, TPEFB, UBF, and TBF fibers. Contact angle is the measure of the wetting property of the surface. Samples measuring 15 mm x 15 mm were prepared. Using 100µL syringe size, each sample was placed on the holder platform of the contact angle setup. Five µL of distilled water was dispensed onto the fiber surface using VCA Optima micropipette (following ASTM D 7334 8 – Standard Practice for Surface Wettability of Coatings, Substrates, and Pigments by Advancing Contact Angle Measurement. The analysis was conducted at the MiNT-SRC Laboratory, Universiti Tun Hussein Onn Malaysia (UTHM), Parit Raja, Johor. Static water contact angle of each sample was determined by analyzing droplet images using advanced software with machine vision capabilities, incorporating 0.5-second trigger delay to capture and calculate the contact angle from live images.

Scanning Electron Microscopy

Analysis of Scanning electron microscope (SEM, JEOL JSM-6380LA) was performed employing concentrated electron beams to scan the surface of the sample and generate high-resolution images. This analysis was conducted at the UTHM's laboratory, in accordance with ASTM D3576. To minimise surface charging during analysis, the sample must be coated with a conductive layer using the JFC-1600 auto fine coater. The sample's front surface was examined in free-rise direction at 10 kV accelerating voltage, and imaging was performed at 30X magnification with 500µm scale, as described by Wang, Lu, Zhou, Lu & Ran (2019).

Thermogravimetric Analysis

Thermal degradation analysis is essential for understanding the fundamentals of thermal stability. Thermogravimetric analysis (TGA) was conducted in this study to determine the thermal degradation of nonwoven untreated and treated UPEFB, TPEFB, UBF, and TBF fiber samples. The analysis was performed using TA Instruments TGA Q500 and TGA 550, according to ASTM E1131 standard for TGA testing. Approximately 10 mg of specimen was placed in a ceramic crucible, and heated from 40 to 700°C at a constant heating rate of 10 °C/min under a nitrogen flow rate of 25 ml/min, following the method outlined by Wang, Lu, Zhou, Lu & Ran (2019).

Fourier-Transform Infrared Spectroscopy

Fourier-transform infrared spectroscopy (FTIR) is a fast, non-destructive, and cost-effective technique used to identify functional groups present in nonwoven untreated and treated UPEFB, TPEFB, UBF, and TBF fiber samples using the Agilent Cary 630 FTIR spectrometer, in accordance with ASTM D6342-12 - Standard Practice for Polyurethane Raw Materials: Determining Hydroxyl Number of Polyols by Near Infrared (NIR) Spectroscopy. The objective was to identify the chemical functional groups present in both untreated and treated samples. Spectra results were acquired within the wavelength range from 750 to 4000 cm^{-1}, with a maximum resolution of 4 cm^{-1}.

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Results

Hydrophobic Properties Of Fibers By Water Contact Angle

The water contact angle (WCA) is a measure of the wettability of a surface, with higher angles indicating greater hydrophobicity or water repellence. In this context, a higher WCA suggests that the surface is less prone to wetting by water droplets, indicating enhanced hydrophobic properties. Table 1 compares the WCA values between UBF and TBF fibres, revealing a significant increase in hydrophobicity following treatment. Specifically, the WCA of UBF fiber was measured at 106.05°, whereas the WCA of TBF increased to 128.35°. This substantial increase in WCA indicates that the treatment process has effectively enhanced the hydrophobicity of bamboo fibers, making them more water-repellent.

Similarly, for PEFB fibers, the treatment has led to a notable increase in WCA. The WCA of UPEFB fibers was recorded at 83.05°, whereas the WCA of TPEFB fibers significantly rose to 130.95°. This considerable improvement in hydrophobicity highlights the treatment efficacy in amplifying the water-repellent nature of PEFB fibers. The observed increase in WCA values post-treatment aligns with findings from previous research in the natural fiber composites field. A study by Danish (2022) demonstrated that surface modification treatments, such as silane or coupling agent treatments, can introduce hydrophobic functional groups onto the surface of natural fibers, thereby reducing their affinity for water molecules. This reduction in water affinity leads to an increase in WCA values, as evidenced by the results obtained in our study.

Furthermore, the significant difference in WCA values between treated BF and PEFB fibers compared to their untreated counterparts highlights the effectiveness of the treatment method in imparting hydrophobicity to these natural fibers. The treated fibers exhibited WCA values well above 100°, indicating a high degree of water repellency and suitability for applications requiring moisture resistance, such as outdoor construction materials or composite reinforcements for marine environments. The surface treatment enhances the hydrophobic properties of both BF and PEFB fibers. The substantial increase in WCA values post-treatment indicates a significant improvement in water repellency, which is crucial for various industrial applications where moisture resistance is desired. These findings support the advancement of natural fiber composites for the development of sustainable materials with enhanced performance characteristics.

The WCA results of both nonwoven untreated and treated BF and PEFB fibers with silane coupling agent revealed hydrophobic properties. Previous research reported by Wie, Kim, & Kim (2020) suggested that the WCA between 90° and 150° indicates better hydrophobic properties for the surface materials. The result exhibited that the WCA of treated BF with silane coupling agent was 128.35° compared to untreated BF of 106.05°. The WCA of the TBF fiber elevated up to the highest value of 17.4 % for hydrophobic owing to the presence of methyl groups from the applied treatment. In contrast, UPEFB exhibited WCA of 83.5°, indicating the hydrophilic property of the materials, emphasizing the effectiveness of the treatment in altering the surface fiber. Subsequently, treated PEFB demonstrated an increased WCA of 130.95°, signifying a notable enhancement in WCA resistance with the following alternative fiber material in insulation application. This study aligns with previous findings indicating that treatment with a silane coupling agent leads to an increase in WCA, associated with the presence of silane functional groups, particularly Si-O-Si and Si-O-C groups (Siy et al., 2020; Mohammed, Ansari, Pua, Jawaid, & Islam, 2015).

Table 1: WCA results of untreated and treated BF and PEFB

Sample	Water contact angle (°)
UPEFB	83.05
TPEFB	130.95
UBF	106.05
TBF	128.35

These findings indicated that the silane coupling treatment process effectively mitigated the hydrophilic of nonwoven BF and PEFB but also improved the overall water resistance of the other fibers. It was also demonstrated that the silane coupling agent treatment had a considerable impact on increasing the WCA of BF and PEFB consistently. The treatment also led to increased WCA from hydrophilic to hydrophobic properties, emphasizing the silane coupling agent's effectiveness in altering the surface properties of the fibers. Hence, this possibility suggested that nonwoven BF and PEFB fibers treated with silane coupling agents exhibited excellent potential to repel water absorption for building applications.

SEM

The SEM images of nonwoven UPEFB, TPEFB, UBF, and TBF fibers treated with silane coupling agent are depicted in Fig. 1. PEFB fiber displayed predominantly consistent cylindrical shapes, while BF fiber showed more rectangular shapes. As displayed in Figure 1 (a) and 1(b), the untreated BF strands were closely aligned, and the surface was clear and smooth. It should be noted that the untreated BF fiber surface was covered with a layer of materials, which may consist of lignin, pectin, and other impurities (Wang et al., 2019; Faruk et al., 2012). Generally, BF constitutes cellulose (26-65%), hemicellulose (30%), and lignin (5-31%). In contrast, PEFB has a complex lignocellulosic material consisting of approximately 41.3%-46.5% cellulose (Suprantoet al., 2014),16.2%-46.5% cellulose (Supranto et al., 2014; Ferre et al., 2011), hemicelluloses of 25.3% - 33.8% (Supranto et al., 2014), and lignin of 19%-32.5% (Supranto et al., 2014; Fere et al., 2011; Kumar, Ganguly, & Purohit, 2023).

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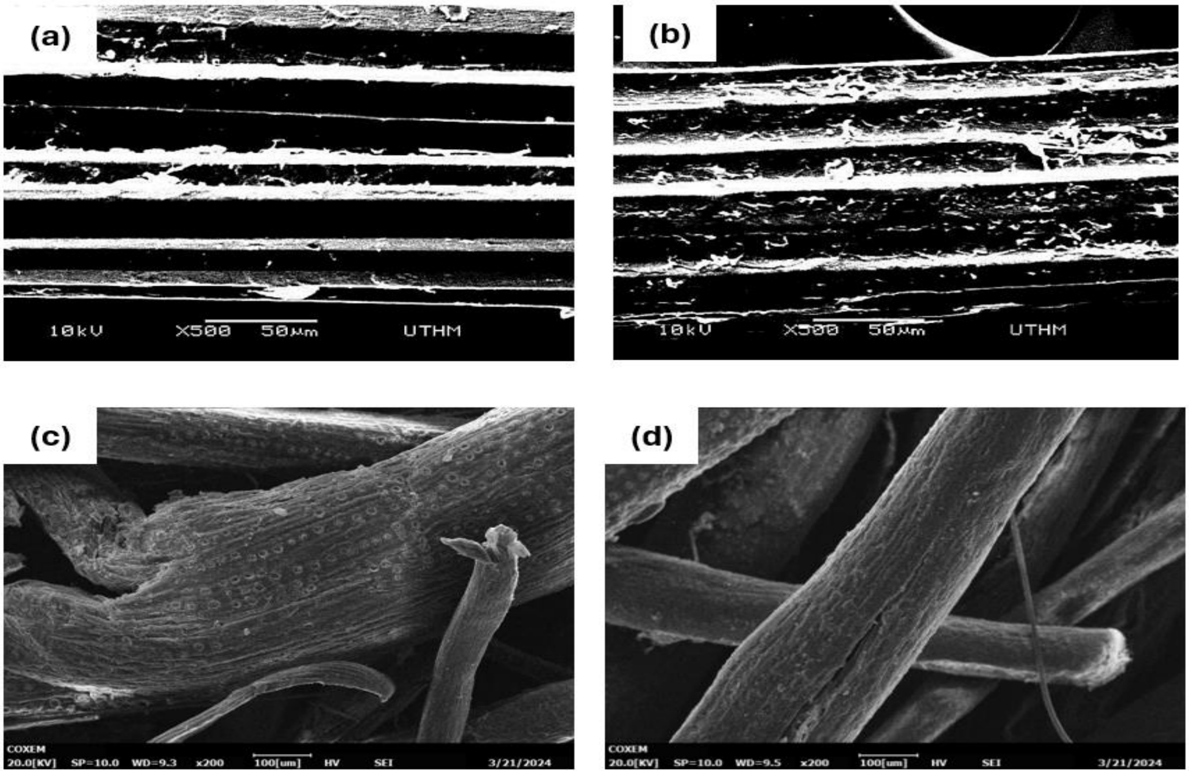

Fig. 1. Morphological Images of (a) Untreated UBF, (b) Treated TBF, (c) Untreated UPEFB, and (d) Treated TPEFB

From the SEM images, it was evident that the surface of the BF fiber showed an enlarged diameter, and the distance of the surface expanded from 0.051 mm to 0.062 mm after silane coupling agent treatment. Meanwhile, the surface area of PEFB fiber demonstrated the opposite characteristic from BF fiber findings. In Figure 1(c) and 1(d), the untreated PEFB fibers showed an axial length of 0.402 mm, and the treated PEFB was 0.246 mm. This revealed that the physical appearance of the surface of the BF fiber was smaller than PEFB fiber. Thus, this indicated that the surface area of the fiber significantly influenced the water absorption performance of the fiber. As referred to in Table 1, the WCA of PEFB was higher than BF fiber. This outcome suggests that the BF fiber treated with the silane coupling agent influenced its axial length of the fiber surface area. This finding also suggests that the increased surface area may effectively repel water absorption, making it suitable for insulation applications.

Thermal Degradation

The thermal properties and weight loss of UPEFB, TPEFB, UBF, and TBF fibers were analyzed by TGA. The TG thermograms are presented in Figures 2 and 3, whereas the TGA results are listed in Table 1. They revealed that the onset of degradation (Ts) of the treated sample shifted to higher temperatures. From Figure 2, it was also explicit that the treatment fiber exhibited homogeneous dispersion and greatly affected the phase behavior of the fiber treated by the silane coupling agent, as previously discussed in the SEM analysis. According to Shenoy Heckadka et al. (2023), thermal decomposition begins above 80 ◦C and 85 ◦C for treated bamboo and PEFB fibers, with their untreated counterparts beyond 90 ◦C. However, in this study, the untreated fibers showed the expected weight loss, while the weight loss of treated bamboo significantly increased, as observed in the thermogram.

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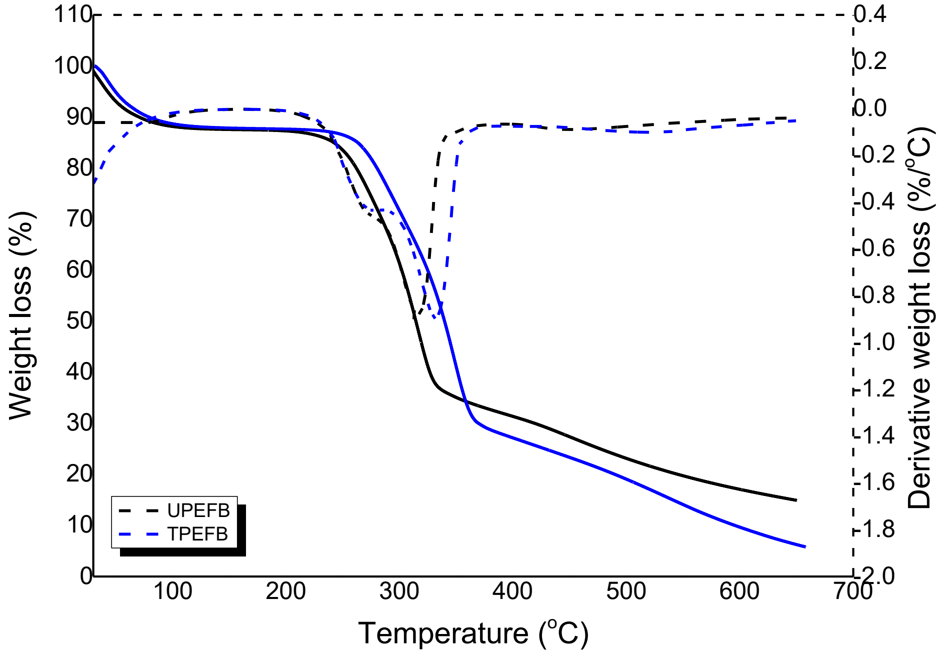

Fig. 2. Thermogravimetric And Derivative Weight Loss Curves Of Untreated And Treated PEFB

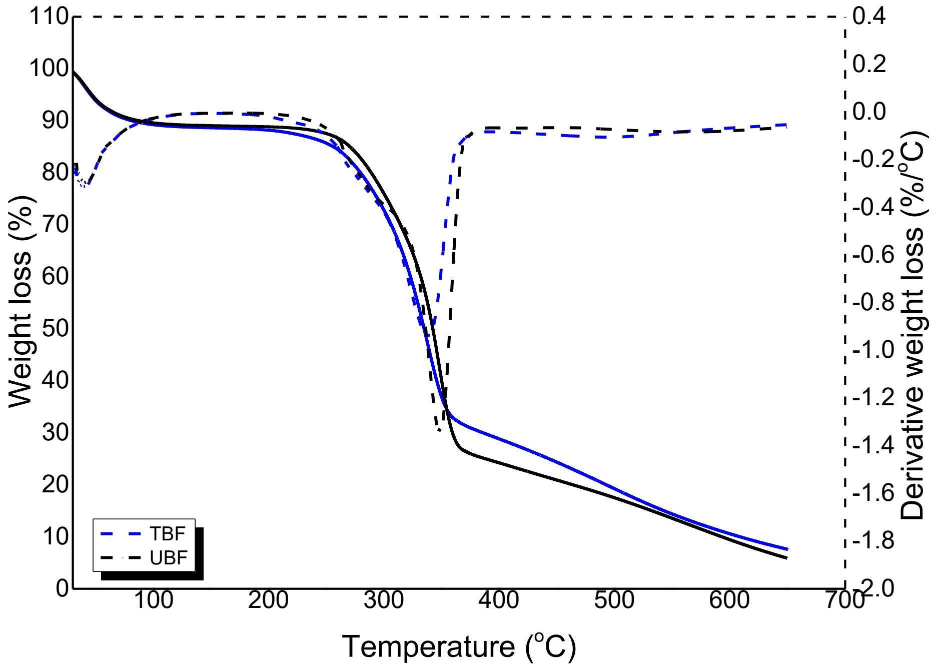

Fig. 3. Thermogravimetric And Derivative Weight Loss Curves Of Untreated And Treated BF

This reflection might be attributed to a good interface between the treated sample of bamboo and PEFB, governed by the incorporation of surface treatment materials, as explained in the SEM analysis and tensile properties sections. The treatment of different fibers demonstrates a synergistic effect, leading to uniform heat distribution during thermal testing. With treatment,

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the interface between bamboo and PEFB fibers crosslinking had been enhanced, delaying the unfavorable enthalpy conditions associated with increased molecular motion, leading to enhanced thermal stability. In contrast, untreated bamboo and PEFB fibers depicted weakened phase separation and crosslinking owing to insufficient surface treatment of the fiber. Although thermal stability indicated improvement under 1.5% and 2.0% loads, it was insignificant. In this study, treated bamboo and PEFB exhibited better thermal stability than the untreated. Notably, 1% silane agent could favor improved thermal stability of treated bamboo and PEFB.

Table 1 displays the critical points of TGA for both treated and untreated PEFB and bamboo fibers. The analysis was conducted under a nitrogen environment with 10 ◦C/min heating rate. In this context, T_S denotes the onset degradation temperature, T_m signifies the maximum decomposition temperature, and T_{50} indicates the temperature at which the composite experienced 50% mass loss. Table 1 presents key thermal properties of both untreated and treated PEFB and bamboo fibers. When comparing the untreated and treated bamboo fibers, there was a noticeable decrease in T_S, T_{50}, and T_m values after treatment. For instance, T_S was reduced from 263°C to 246°C, T_{50} decreased from 343°C to 338°C, and T_m dropped from 368°C to 356°C after treatment. It suggests that the silane coupling agent treatment process has effectively amplified the thermal stability of bamboo fibers, leading to a delay in the onset of degradation and lowering the temperatures at which significant mass loss occurs.

Table 1 Critical Points Of Thermogravimetric Analysis (TGA) For Treated And Untreated PEFB And BF.

Sample	Onset temperature, Ts (°C)	50% decomposition temperature, T50 (°C)	Maximum degradation temperature TM (°C)	Char residue (wt%)
Untreated BF	263	343	368	6.79
Treated BF	246	338	356	5.06
Untreated PEFB	233	329	354	7.06
Treated PEFB	244	335	357	6.16

Similar trends can be observed in the case of PEFB fiber. The treated PEFB fibers exhibited lower T_S, T_{50}, and T_m values compared to their untreated counterparts. Specifically, T_S decreased from 233°C to 244°C, T_{50} decreased from 329°C to 335°C, and T_m decreased from 354°C to 357°C after treatment. These results indicated that the treatment of PEFB fibers has also contributed to improving their thermal stability, as evidenced by the shift in critical degradation temperatures to lower values. The observed decrease in critical temperatures post-treatment aligns with findings from previous research in the natural fiber composites field. Study by Karvanis, Rusnáková, Krejčí, & Kalendová (2021) demonstrated that chemical treatments, such as alkali or silane treatments, can lead to the removal of impurities and hemicellulose from natural fibers, resulting in a more homogeneous and thermally stable fiber structure. This removal of impurities reduces the tendency of fibers to undergo thermal degradation at higher temperatures, thus lowering the critical degradation temperatures observed in TGA analysis, as seen in the results.

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Furthermore, the decrease in char residue percentages for both treated bamboo and PEFB fibers compared to their untreated counterparts was significant. Char residue refers to the percentage of material remaining after complete thermal degradation and is indicative of the material's ability to form a stable carbonaceous residue. The decrease in char residue percentages after treatment suggests that the treated fibers undergo more thorough degradation, leaving behind less residual carbonaceous material. This finding is in agreement with previous studies (Marchi et al., 2023; Guan et al., 2015), which have shown that chemical treatments can alter the chemical composition and structure of natural fibers, affecting their char-forming ability during thermal decomposition.

FTIR

Fig. 4 displays the FTIR spectra of nonwoven untreated and treated BF and PEFB fibers with silane coupling agents. The absorption peaks were observed at various wavenumbers corresponding to specific functional groups of BF and PEFB fibers. The peaks of the nonwoven untreated and treated BF and PEFB fibers were observed at 3400, 2906, 1620, and 1150–1050 cm^{-1}, which were ascribed to the stretching vibrations of –OH groups, C–H groups, H–C–H rings, and C–O groups in cellulose, respectively. For instance, peaks observed at 2851 and 897 cm^{-1} corresponded to the stretching and rocking vibrations, respectively, of the C–H groups in cellulose. In addition, the peak at 1238 cm^{-1} indicated stretching of C–C groups in hemicellulose, while the peaks at 1639 and 1512 cm^{-1} were related to the stretching of C=O and C=C bonds of aromatic ring in lignin. These findings were aligned with previous research on fiber (Wang et al., 2019).

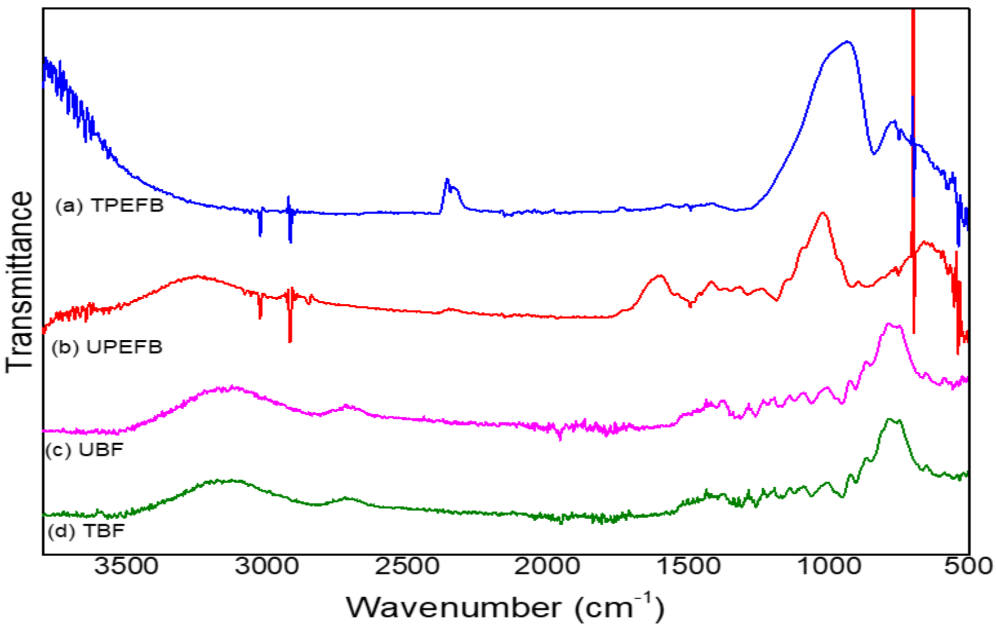

Fig. 4. FTIR Of Untreated Bamboo Fiber, Treated Bamboo Fiber, Untreated PEFB, And Treated PEFB

The nonwoven untreated and treated BF and PEFB fibers exhibited antisymmetric stretching from 975 to 990 cm-1 wavenumbers, which could represent ether bonds. The stretching absorbance in the spectra is attributed to the ester bond resulting from the complete cleavage of hemicellulose (Wang et al., 2020). Additionally, lignin peaks were detected within the

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wavenumber range of 990 to 1090 cm^{-1}. Interestingly, the peak patterns in untreated BF and PEFB fiber remained similar. On the other hand, after treatment, the deformation of Si-O-C bond stretching vibrations was observed from 1000 to 1100 cm^{-1}.

In the treated BF fiber, prominent peaks appeared in the range of 3010 to 3040 cm^{-1} after treatment, indicating Si-O-Si and Si-O-C stretching vibration. They could represent new chemical bonds formed between silane coupling-treated BF and PEFB. The peak observed at 3300 cm^{-1} corresponded to the O-H hydrogen bonding stretching in cellulose in the treated BF and PEFB. Prominent peaks were observed in the treated BF and PEFB fiber from 3010 cm^{-1} to 3040 cm^{-1} after treatment, which could be attributed to Si-O-Si and Si-O-C stretching vibrations.

However, peaks assigned to the C=C stretching bond in the range of 1638 to 1648 cm^{-1} were absent in this FTIR analysis. The reason was presumably due to the low concentration of silane coupling agent, which was insufficient to generate peak changes in the FTIR spectra. In addition, the signal around 1540 cm-1 may represent the C=O and C-N stretching vibrations of the urethane linkage. Similar to the study by Abidin et al. (2025), the silane coupling-treated BF and PEFB showed better bonding than the untreated fiber. Thus, the silane coupling agent treatment is deemed to improve the crystallinity of the cellulose in PEFB.

Conclusions

In conclusion, this study examined the physical characterization, including WCA, surface morphology, thermal decomposition behavior, and functional groups of untreated and treated bamboo fiber (BF) and palm empty fruit bunch (PEFB) fiber. The results shed light on the effectiveness of surface treatment techniques in enhancing the properties of natural fibers. A significant increase in WCA values was observed in both silane coupling-treated BF and PEFB fibers, indicating improved hydrophobicity. The treated fibers exhibited WCA well above 90°, suggesting a high degree of water repellence and suitability for applications requiring moisture resistance. Also, the surface morphology of both BF and PEFB fibers displayed alterations in surface diameter and axial length, with treated fibers demonstrating characteristics that successfully improved water repellence. Moreover, the presence of C-O, C=O, and OH groups was associated with cellulose, hemicellulose, and lignin in both untreated and treated fibers. After treatment, the presence of peaks corresponding to Si-O-C and Si-O-Si stretching vibrations indicated that new chemical bonds were formed, contributing to enhanced bonding and stability. Overall, the findings highlight that the silane coupling agent treatment was effective in improving the hydrophobicity, morphological characteristics, thermal stability, and functional properties of BF and PEFB fibers. These enhanced properties make the treated fibers promising BF and PEFB fibers for various industrial applications, particularly as insulators.

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