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Morphological, TGA, and FTIR on Rigid Polyurethane Composite Laminated with Untreated and Treated Bamboo Fiber Roof Insulation

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ABSTRACT

The performance of roof insulation such as polyurethane decreased due to problems such as insufficient absorption and poor thermal insulation performance, especially during rainstorms. The aims of this study are to investigate the physical property and its potential reinforced material such as rigid polyurethane doped with treated and untreated bamboo fiber composite (RPU-BF) at different ratios of 0, 25, 30, 35, and 40% of bamboo fibers as an insulation material for roof applications. The bamboo fibers were treated by using silane coupling agent treatment. The rigid polyurethane composite samples were prepared and then laminated bamboo fiber to overcome the sound problem in roofs. The physical characterization was investigated by Water Contact Angle (WCA), the morphological by Scanning Electron Microscopy (SEM), Thermo-gravimetric Analysis (TGA), and Fourier Transform Infrared Spectroscopy (FTIR) Analysis. The results showed that the treated bamboo fiber had a 192.5° water contact angle as a super hydrophobic property due to the presence of the chemical bonds Si-O-Si and Si-O-C in the silane coupling agent treatment. The morphology showed that 30% ratios of RPU-BF-T30 give the smallest pore diameter size. The peak of thermal degradation temperature of untreated and treated bamboo fiber was increased from 320°C to 350 °C with a weight loss of 80% to 50%. The treated bamboo fiber exhibited peaks at 3010–3040 cm^{-1} were associated with stronger Si-O-Si bonding, indicating the formation of new chemical bonds between bamboo fiber and silane coupling agent due to the ester bond from the cellulose, lignin, and hemicellulose. Thus, there was a similar trend peak in the functional chemical group in the FTIR spectrum of the RPU-BF composite. This result shows that RPU-BF composite had the potential of the optimum ratio of bamboo fiber as an insulation material for local communities and beneficial to the bamboo industry.

Keywords:
Bamboo fiber; rigid polyurethane; physical characterization

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1. Introduction

Rigid polyurethane (RPU) has a number of desirable characteristics, including excellent mechanical properties, high weather ability, low thermal conductivity, low density, and excellent damping abilities by Borowicz & Isbrandt [1]. In addition, RPU has a variety of uses in the automotive, packaging, and construction industries by Zieleniewska *et al.*, [2]. Therefore, reinforcement polymer with different fillers is a widely used method to enhance foam properties by Widya & Macosko [3]. RPU is produced via exothermic reactions between alcohols and two or more reactive hydroxyl groups (-OH) per molecule (dials, trials, polyols) and isocyanates that have more than one reactive isocyanate group (-NCO) per molecule of diisocyanates or polyisocyanates by lvdre *et al.*, [4]. The geometrical anisotropy of cells is translated into the anisotropy of the mechanical characteristics of the foams by Andersons *et al.*, [5]. Adding various functional particles or reinforced fibers such as bamboo fiber into the RPU foams has been an efficient method to promote the insulation properties of RPU foams studies by Chen & Jiang [6].

Bamboo is a cylindrical, typically hollow, lightweight, and functionally graded material with ideal properties for building truss elements, which are commonly used in roof insulations [7]. Wang *et al.*, [8] identified that the anatomical properties of bamboo are important as they influence mechanical properties, preservative absorption, and the properties of end products, especially pulp and paper industry. Bamboo does not necessitate replanting after harvesting as its extensive root network constantly sprouts new shoots that almost zoom upwards, absorbing sunlight and greenhouse gases, and converting them to new green plants by Kumar *et al.*, [9]. Bamboo also has distinct properties, such as anti-UV radiation, antibacterial, breathable, cool soft handle, and so on. It has greater moisture absorption due to the numerous micro gaps and micro holes in the cross-section by Prakash [10].

In addition, the bamboo fiber has a small number of surface hydroxyl groups and a large particle size, which limits its ability to participate in the polyurethane foaming reaction by Qiu *et al.*, [11] and it contains a hydrophilic nature that may limit its applications. Many chemical treatments are performed on bamboo fiber such as alkalization by Sugiman *et al.*, [12], peroxide by Abd Halip *et al.*, [13], and silane by Siy *et al.*, [14]. These treatments can successfully improve the fiber from hydrophilic to superhydrophobic with a water contact angle measurement range of more than 150°.

RPU also has great potential for sound absorption by Zhu *et al.*, [15], and is used to make the majority of acoustic foam panels in roof insulation materials especially on laminated insulation by Sharma *et al.*, [16]. Two composite face sheets and a core form a composite laminated structure by Kondratiev *et al.*, [17]. Laminated structures can be used as components and provide a variety of advantageous mechanical, chemical, and physical properties in addition to being lightweight by Junaedi *et al.*, [18]. For laminated structure fiber doped polymers such as glass-fiber have become an option due to their excellent properties in terms of strength, stiffness and light weight but interfacial bonding between the facing skins and core is also found fail in the interfacial due to transverse shear force at a lower force by Fatima *et al.*, [19].

In this study was characterized bamboo fibers treated with silane coupling agent to enhance the surface property from hydrophilic to superhydrophobic and produced Rigid Polyurethane Composite Laminated with Untreated and Treated Bamboo Fiber Roof Insulation. The laminated method is lightweight, and its mechanical properties make it suitable for use in a wide range of industries and can be used for a variety of decorative and building purposes by J. Li *et al.*, [20]. The laminated construction development will improve the qualities of polyurethane foam as well as laminated composite panels by integrating natural fiber with polyurethane foam as a core by Khan & Acar [21]. Hence, due to the potential of modifying bamboo fiber to obtain superhydrophobic properties and concerned with RPU composite in roof insulation, the ideal of this study is to characterize the bamboo fiber treated with a silane coupling agent and produce Rigid polyurethane doped bamboo fiber (RPU-BF) composites for use in laminated roof insulation applications.

2. Methodology

Bamboo fiber was obtained from HangTerra Bamboo Sdn Bhd. The diameter of the bamboo fiber was prepared in the range of <0.2 to 0.3 mm, and extracted from the culm strip to the fiber, and the fiber was cut within 30 mm in length. RPU foams were produced by mixing polyol and isocyanate which were purchased from Scientifik Bersatu (M) Sdn Bhd. In the sample, the polyol-PMDI ratio was 100/100, with polyol viscosity at 260 mPa and isocyanate viscosity at 20°C [22].

2.1 Preparation of Untreated and Treated Nonwoven Bamboo Fiber

2.1.1 Treatment bamboo fiber treated silane coupling agent

The bamboo fibers were treated with a silane coupling agent 371890 (ALDRICH) from Dwicitra Resources. An ethanol-to-distilled water weight ratio of 80:20 was mixed with 0.1 g 3-aminopropyl (diethoxy) methyl silane hydrolyzed, and then the mixture was stirred at a constant pace until it was completely dissolved. Then, 10 g of bamboo fibers were added into the solution and were continually agitated (silane-filler ratio of 1: 100). The bamboo fibers were soaked in silane solution for 3 hours before their pH was adjusted to 4.00 using the modified method of acetic acid. After that, the bamboo fibers were oven-dried for 24 hours at 80°C [22].

2.1.2 Preparation of nonwoven bamboo fiber

The untreated and treated nonwoven bamboo fibers were prepared using a needle-punching machine. The thickness of the untreated and treated nonwoven bamboo fibers appeared to be middling at 3 mm. The bamboo fibers were loaded at the opening, and the blower and beater were opened simultaneously. The bamboo fibers were equally fed into the carding machine to make the fiber web. The bamboo fiber web was transformed into nonwoven bamboo fiber by a needle punching machine with approximately 2000 pieces/M2 needle and a 300 m/min speed.

2.1.3 Fabrication of rigid polyurethane foams composite bamboo fiber

Fabrication of RPU-BF composite began with the preparation of the polyol and isocyanate ratio of 1:1. RPU-Pure composite was fabricated using a one-shot process, where polyol and isocyanate were mixed inside the mould [23]. The polyol was stirred by a mechanical stirrer with a rotor speed of 400 rpm until the colour turned white for 3 minutes. Then, the bamboo fibers were added with isocyanate and stirred for 60 seconds until homogenous. Then, the polymerization process began, resulting in a urethane bond produced by polyether polyol and isocyanate in greater molecular weight. Upon the completion of the expansion, the RPU-Pure composite was held inside the mould for 24 hours for the polymeric bond to be stable and cut to the specified size for characterization.

3. Physical Characterization of Untreated and Treated RPU-BF

3.1 Water Contact Angle

The untreated and treated nonwoven bamboo fiber were prepared in dimensions of 15 mm x 15 mm, and double-sided tape was placed on the glass slide. The fiber sample was placed on the contact angle setup holder platform substrate with a syringe size of 100 µL. Then 5 µL of distilled water was applied to the fiber surface areas with a micropipette by VCA Optima by ASTM D 7334 8 at the MiNT-SRC laboratory, Universiti Tun Hussein Onn Malaysia (UTHM), Parit Raja, Johor. The static water contact angles of untreated and treated nonwoven bamboo fibers were determined from droplet capture images using Image J software capable of measuring the contact angle of a water droplet calculated from a live image with advanced machine vision capacity trigger delay of 0.5 sec.

3.2 Morphological Characterization

Scanning Electron Microscopy (SEM) is a type of electron microscope that uses a concentrated beam of electrons to scan the surface of a sample to produce images. In this study, SEM (JEOL JSM-6380LA) analysis was conducted at the Material Characterisation Laboratory, UTHMs, Parit Raja, and Johar according to ASTM D3576. To prevent electrical charge collection, the test samples were gold-coated. The front surface of the untreated and treated bamboo fibers was examined and scanned in a free-rise direction at the accelerating voltage of 10 kV, followed by 100 µm at 500x magnification.

3.3 Thermal Characterization

Thermo-gravimetric Analysis (TGA) is an analytical technique for determining material thermal stability and the proportion of volatile components by monitoring the weight change that occurs as a sample is heated at a constant rate. TGA 550 TA Instrument Trios Discovery Series and ASTM El 131 standard methods were used to determine the thermal characteristics of the untreated and treated bamboo fibers that were conducted at Process Control Laboratory, UTHM, Pagoh Campus, Johar. The untreated and treated bamboo fibers were heated from 40°C to 700°C in a nitrogen environment at a rate of 10 °C/min with a flow rate of 25 ml/min.

3.4 Functional Group Characterization

Fourier-Transform Infrared Spectroscopy (FTIR) analysis was conducted using Agilent Technologies Cary 630 FTIR by ASTM D6342-12 to determine the chemical functional groups of untreated and treated bamboo fibers. This analysis was conducted at the Chemistry Analytical laboratory at UTHM, Pagoh Campus. The measurement was performed with a maximum resolution of 4 for scanning the 650 to 4000 cm^{-1} wavelength range.

4. Result and Discussion

4.1 Physical Characteristics and Morphology of Untreated and Treated Nonwoven Bamboo Fiber

4.1.1 Water contact angle

Figure 1 shows the results of the water contact angle of the untreated and treated bamboo fibers with a silane coupling agent. A greater water contact angle between 120° and 150°indicates better hydrophobicity for bamboo fibers by Altangerel *et al.*, [24]. The water contact angle of untreated bamboo fiber was 160.2°, while the average water contact angle of treated bamboo fiber was slightly higher at 192.5°. The increment of treated bamboo fiber was found to be 22.2%, which was the highest value for superhydrophobic due to the existence of methyl groups from the applied treatment. Treatment with a silane coupling agent enhances the water contact angle based on the silane functional group by Wie *et al.*, [25], such as Si-O-Si and Si-O-C groups by Siy *et al.*, [14].

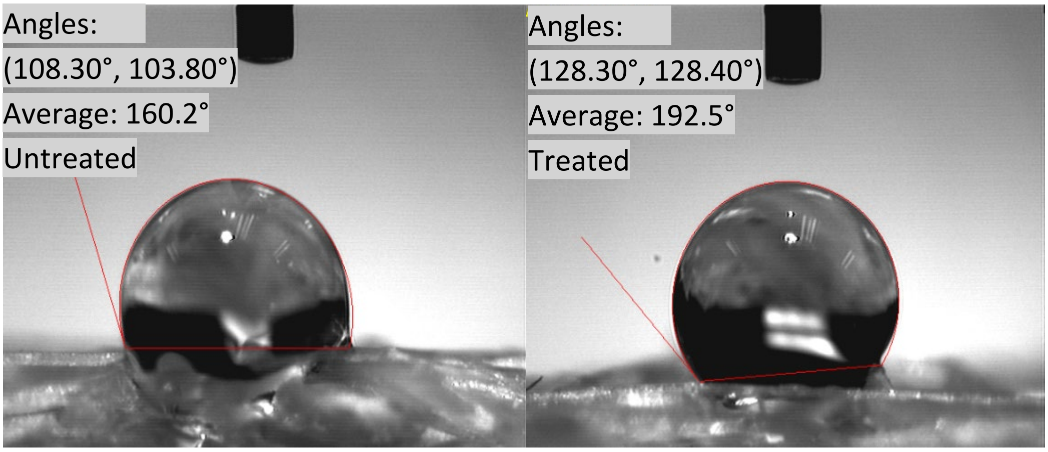


Fig. 1. Water contact angle nonwoven untreated and treated bamboo fiber

Moreover, the silane coupling agent treatment destroyed the interaction between bamboo fibers based on the shape and wrinkled surface displayed which could increase specific area as well as expand the effective contact area. Hence, this possibility suggested that nonwoven bamboo fibers treated with silane coupling agents demonstrated outstanding development potential in laminated to repel water absorption suitable for roofing and building applications.

4.1.2 Pore diameter size of rigid polyurethane composite with untreated and treated bamboo fiber

Figure 2 shows the SEM micrographs of RPU composites doped with untreated and treated bamboo fibers. From the observation, there was not much difference between the images captured from the surface perpendicular to the foaming direction. In Figure 2, it can be seen that the RPU-Pure foam has the largest average pore diameter is 0.0688. Based on literature, it has been mentioned that almost all man-made foams are anisotropic by Myers *et al.*, [26].

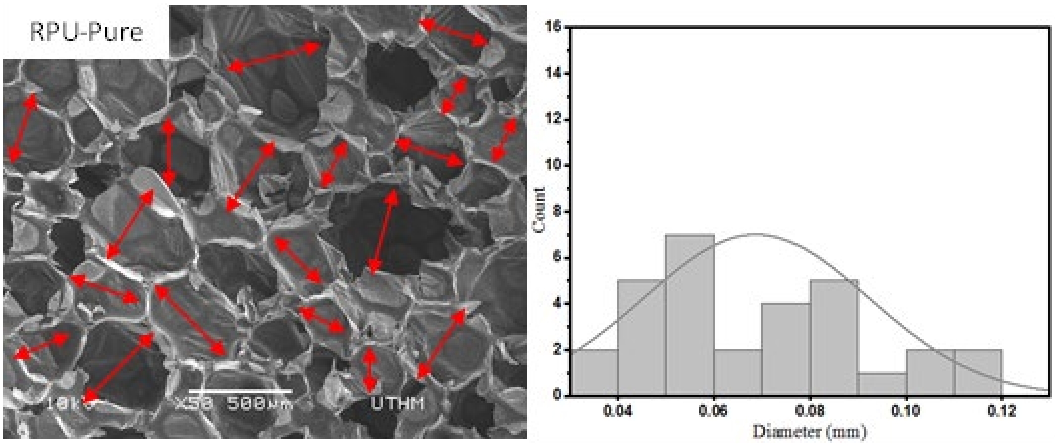
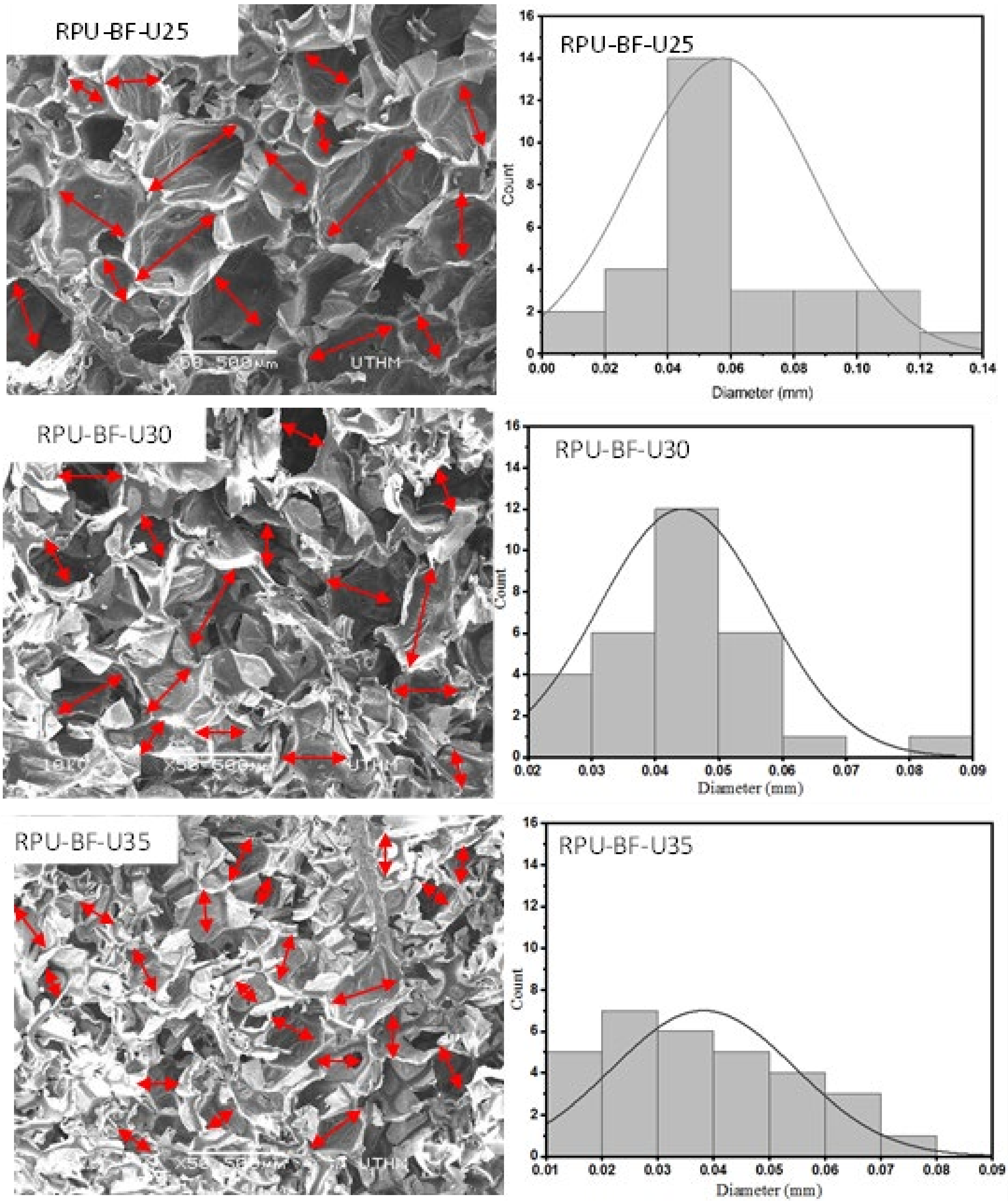


Fig. 2. The morphological structure and diameter pore size of RPU-Pure composites

Based on the SEM micrographs, the RPU composites have an open closed cell structure, and CO_2 was chosen to fill the cells to increase the insulation potential of the foam. Since the bubbles can withstand significant pressure, the foam can be rigid or strong by Defonseka, C., [27]. Compared to the pore structures of the RPU-BF composites, larger cells were found on the RPU-Pure composite structure on surface perpendicular to the foam direction. The RPU-BF composite showed different diameter pores and structures as it contained untreated and treated bamboo fibers as shown in Figures 3 and Figure 4. This can be proven by the greater diameter pore size of 0.0688 mm. Based on the figures, the diameter pore size of the RPU-BF composite become smaller by increasing the percentages of bamboo fibers due to bamboo fiber treatment by Oushabi *et al.*, [28] and Sair *et al.*, [29].



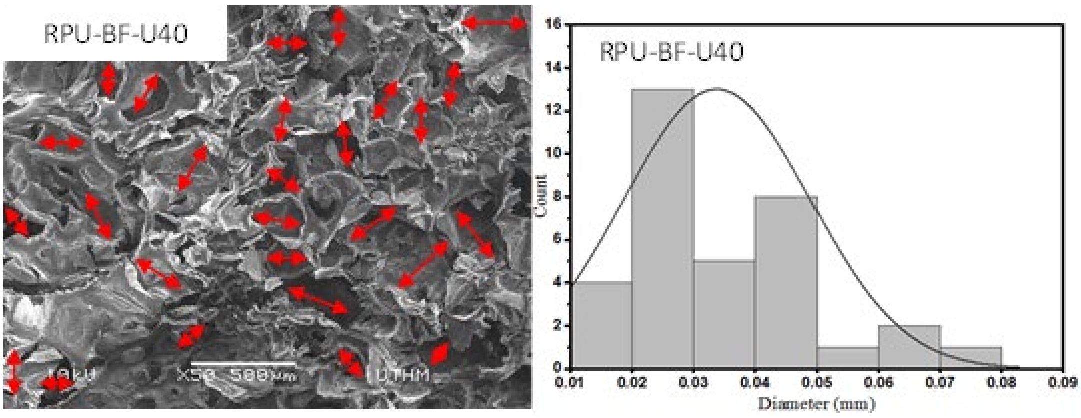
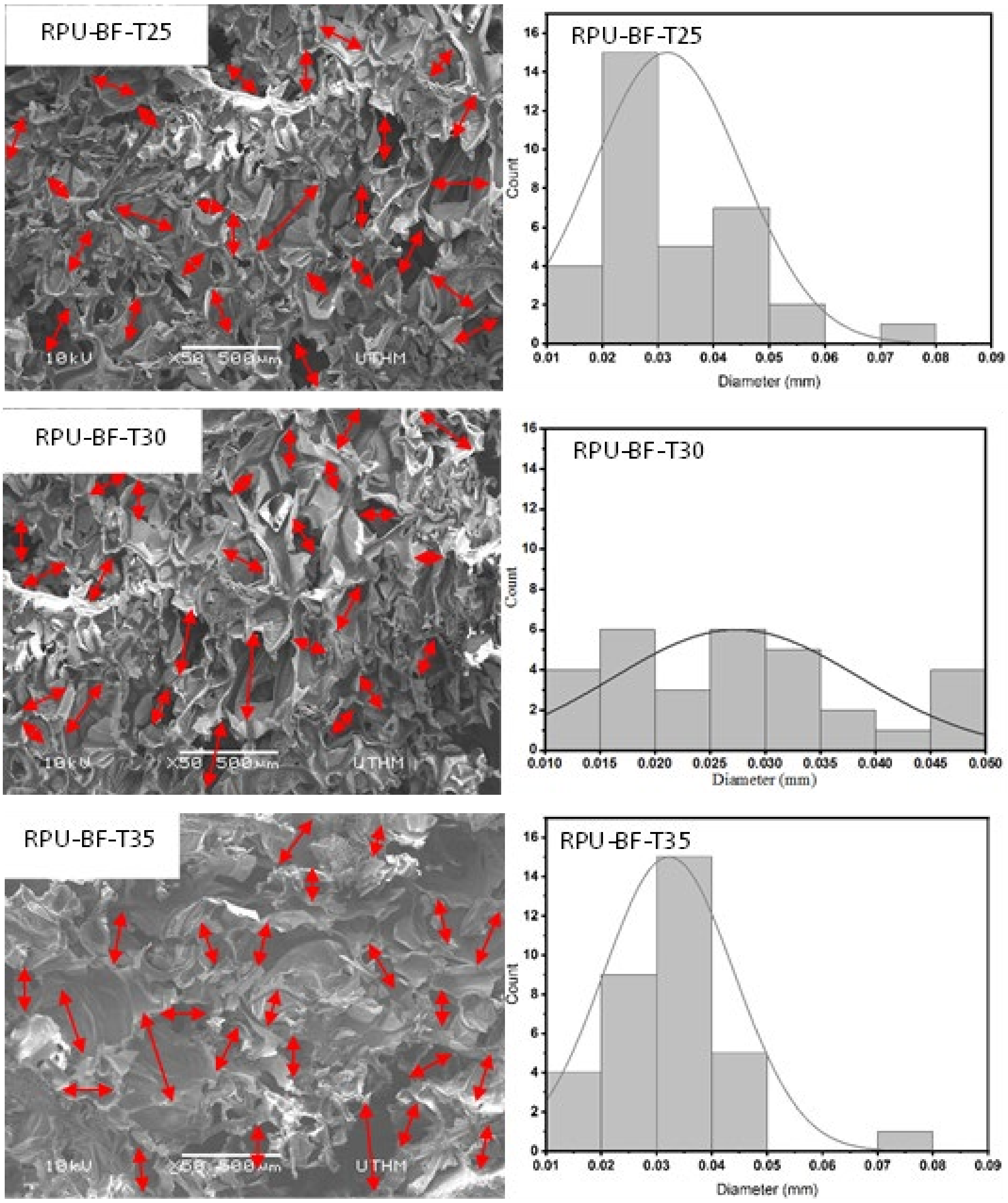


Fig. 3. The morphological structure and diameter pore size of RPU-BF composites with untreated bamboo fiber



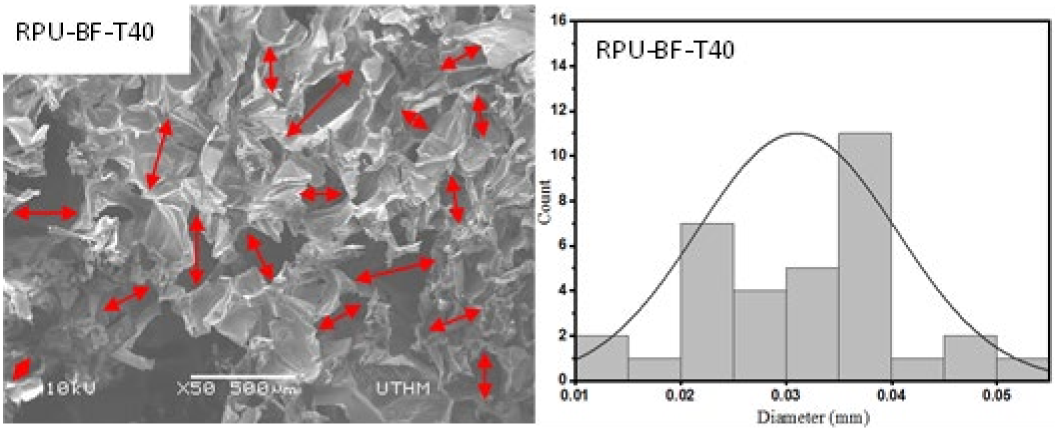


Fig. 4. The morphological structure and diameter pore size of RPU-BF composites with treated bamboo fiber

The average pore diameter sizes for RPU-BF-U25 and RPU-BF-U40 containing untreated bamboo fiber were 0.0575 mm and 0.0338 mm, respectively, as presented in Table 1. Without bamboo fiber, the RPU-Pure foam composite exhibited a maximum pore diameter size of 0.0688 mm. If compared with the RPU-BF composites containing treated bamboo fibers, the pore diameter sizes of RPU-BF-T25 to RPU-BF-T40 were from 0.0317 mm to 0.0310 mm, smaller than those of RPU-BF containing untreated bamboo fibers. The smallest pore diameter size of 0.0274 mm was recorded by RPU-BF-T30 composite among others samples.

Table 1
The diameter pore size of RPU-BF composite

Bamboo Fiber (wt %)	Average pore diameter size (mm)
RPU-Pure	0.0688
RPU-BF-U25	0.0575
RPU-BF-U30	0.0443
RPU-BF-U35	0.0383
RPU-BF-U40	0.0338
RPU-BF-T25	0.0317
RPU-BF-T30	0.0274
RPU-BF-T35	0.0323
RPU-BF-T40	0.0310

Furthermore, the SEM micrographs depicted that larger pore diameter sizes were formed on the cell walls of RPU-Pure composite which may contribute to low sound absorption capability. Moreover, the porosity of the RPU-BF composite can be determined by the number of pores that can lead to lower and higher absorption of sound waves through the RPU-BF composite foams by Choe *et al.*, [30]. Therefore, comparatively, the pore diameter size decreased with an increasing percentage of untreated and treated bamboo fibers.

4.1.3 Thermal analysis of untreated and treated bamboo fiber

The thermal degradation of untreated and treated bamboo fibers was examined by TGA. The weight loss and derivative weight curves of untreated bamboo fiber are shown in Figure 5. The peak of untreated bamboo fiber at temperature ranges from 20°C to 208°C with weight loss of 12% indicates the volatile material and water mixture. This may be corresponded to the evaporation of water by Fernandez *et al.*, [31] and dissolved of moisture of small molecules in the bamboo fibers by Tang *et al.*, [32]

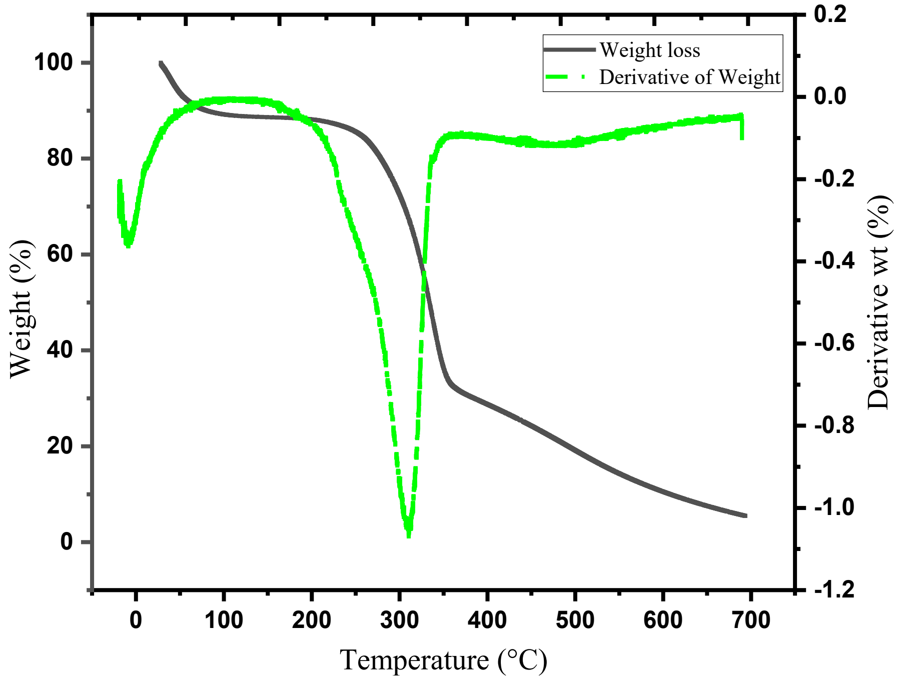


Fig. 5. Thermogravimetric curve weight loss and derivative weight of untreated bamboo fiber

In Figure 5, the thermal degradation peak of untreated bamboo fiber occurred at a temperature of 320°C and shows the weight loss percentage was 80 %. This could be attributed to the decomposition of cellulose in the fibers [32-34]. The peak of thermal degradation temperature impacted the bamboo fibers with an increase in temperature from 208°C to 320°C and a weight loss percentage difference of about 30%. The increasing temperature may be due to the removal of some hydroxyl substances [35]. Meanwhile, the weight loss percentage decreased to 50% at a temperature of 347°C which was attributed to the degradation of lignin in the bamboo fiber and reduction in water mass [36].

Figure 6 shows the the peak of treated bamboo fiber at temperature ranges from 10°C to 265°C with a weight loss of 10% indicating the volatile material and water mixture of fibers. The peak of thermal degradation of treated bamboo fiber showed an increase from 265°C to 350°C, indicating silane coupling agent with bamboo fiber as a result of the reaction which could increase the thermal degradation temperature by Nurazzi *et al.*, [37]. In addition, the thermal degradation peak of untreated and treated bamboo fibers was observed, as summarised in Table 2. This could imply that this cellulose and lignin decomposition by Cavalcanti *et al.*, [38]. The thermal degradation peak was also observed at 350°C with a weight loss of 50% for the treated bamboo fibers, whereas the untreated bamboo fiber indicated a weight loss of 80% at 320°C, 20% difference due to silane coupling agent treatment. It may be due to the reduction in the number of hydroxyl groups that have reacted with the coupling agent by Huang *et al.*, [39]. The treated bamboo fiber displayed the highest thermal degradation temperature at 350°C compared to untreated at 320°C. It signified the surface compatibility and bonding strength of the treated fibers with silane coupling agent were better than the untreated bamboo fiber. However, the treated bamboo fiber exhibited improvement in its thermal degradation temperature. Thus, this material could be incorporated into polymer composites as insulation in building and roofing applications.

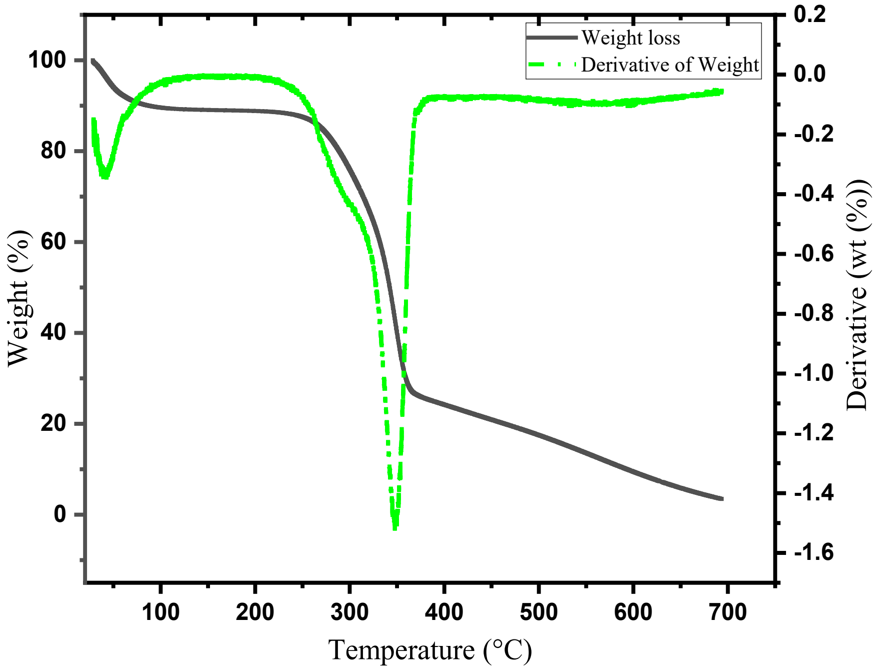


Fig. 6. Thermogravimetric curve weight loss and derivative weight of treated bamboo fiber

Table 2
TGA of untreated and treated bamboo fiber

Bamboo Fibers	Temperature of Weight Loss (°C)	Weight Loss (%)
Untreated	208	12
	320	80
	347	50
Treated	265	10
	350	50
	370	30

4.1.4 Functional group identification analysis of untreated and treated bamboo fiber and rigid polyurethane composite bamboo fiber

Figure 7 depicts the FTIR spectra of untreated and treated bamboo fibers with a silane coupling agent. The untreated and treated bamboo fibers exhibited antisymmetric stretching from 975 to 990 cm^{-1} wavenumbers which may be associated with the ether bonds. The stretching transmittance in the spectra is due to the ester bond of the completely cleaved hemicellulose by Guan *et al.,* [40]. The wavenumber range of 990 to 1090 cm^{-1} represented the peaks of lignin.

For the untreated bamboo fiber, it was observed that the trend of peak was similar to the treated fiber. The transmittance at 1000 to 1100 cm^{-1} after treatment indicated the deformation of Si-O-C bond stretching. For treated bamboo fiber, stronger peaks were observed from 3010 to 3040 cm^{-1} after treatment which could be related to Si-O-Si and Si-O-C stretching vibration. According to Lu *et al.*, [41], new chemical bonds have been formed between the bamboo fibers and the silane coupling agent. However, the transmittance at 1638 to 1648 cm^{-1} assigned to the C=C stretching bond, was not seen in this FTIR analysis. The reason is presumably due to the silane coupling agent being too low in concentration to display all the peak changes in the FTIR spectra. In addition, the signals 1540 cm^{-1}, which are attributed to the C=O and C-N stretching vibration of the urethane linkage, can be used to correspond to the formation of the urethane linkage by Carlos de Haro *et al.*, [42]. Equally to the literature study from Wang *et al.*, [43] the treated bamboo fiber with silane coupling agent shows better bonding than untreated.

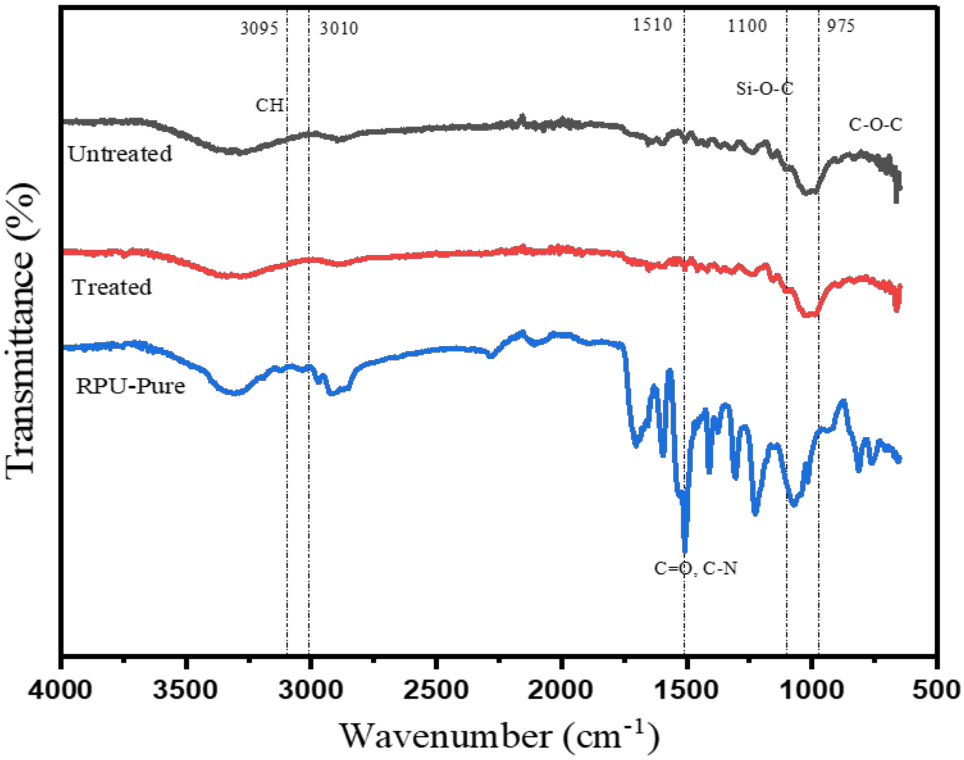


Fig. 7. FTIR spectra of untreated and treated bamboo fibers, and RPU-BF Composite

Figures 8 and 9 depict the FTIR spectra of the RPU-BF composite containing untreated and treated bamboo fibers with different percentages. In Figure 8 it was observed that the N-H group at 3300 cm^{-1} corresponded to the hydrogen-bonded urethane link, which indicated that the isocyanate group reacted with the -OH group to form -NH-COO- by Wang *et al.*, [43]. The transmittance at 3300 cm^{-1} shifted to a lower peak at 3290 cm^{-1} as the concentration -NH-COO- increased, which reflected the formation of hydrogen bonds between the hydroxyl groups in the bamboo fiber source and the N-H group of the rigid or PU foam by S. Zhang *et al.*, [44]. Moreover, the formation of urethane linkages can be confirmed by the appearance of peaks at 1715 cm^{-1} and 1540 cm^{-1}, attributing to C=O and C-N stretching vibration of urethane linkage by Juan Carlos de Haro et al., [42]. The trend of the functional group was similar and did not change much, respectively.

While in Figure 9, the transmittance peaks at 1520 cm^{-1} and 1220 cm^{-1} demonstrated the presence of isocyanurate, which was formed by the reaction between isocyanate and urethane groups by Li *et al.*, [45]. The peak of transmittance at 1702 cm^{-1} in the carbonyl region was a response to the C-O stretching from ketone, aldehyde, and ester groups by Gomez Serrano *et al.,* [46], Yu *et al.*, [47]. Hence, the RPU-BF composite had the transmittance peaks at 1410 cm^{-1}, 1310 cm^{-1}, and 1080 cm^{-1} corresponding to the stretching vibration of the intermolecular hydrogen attraction at RPU-BF-T35 composite the bending of –OH and the stretching vibration of C-O-C ether by Hussin *et al.*, [48].

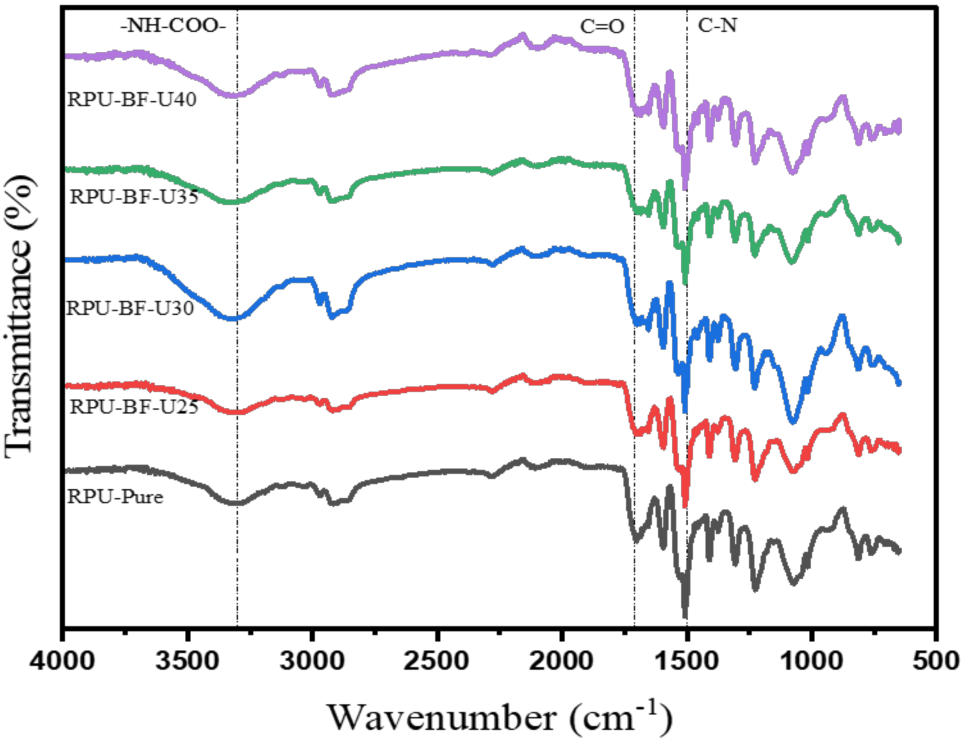


Fig. 8. FTIR spectrum of RPU-BF composite untreated bamboo fiber

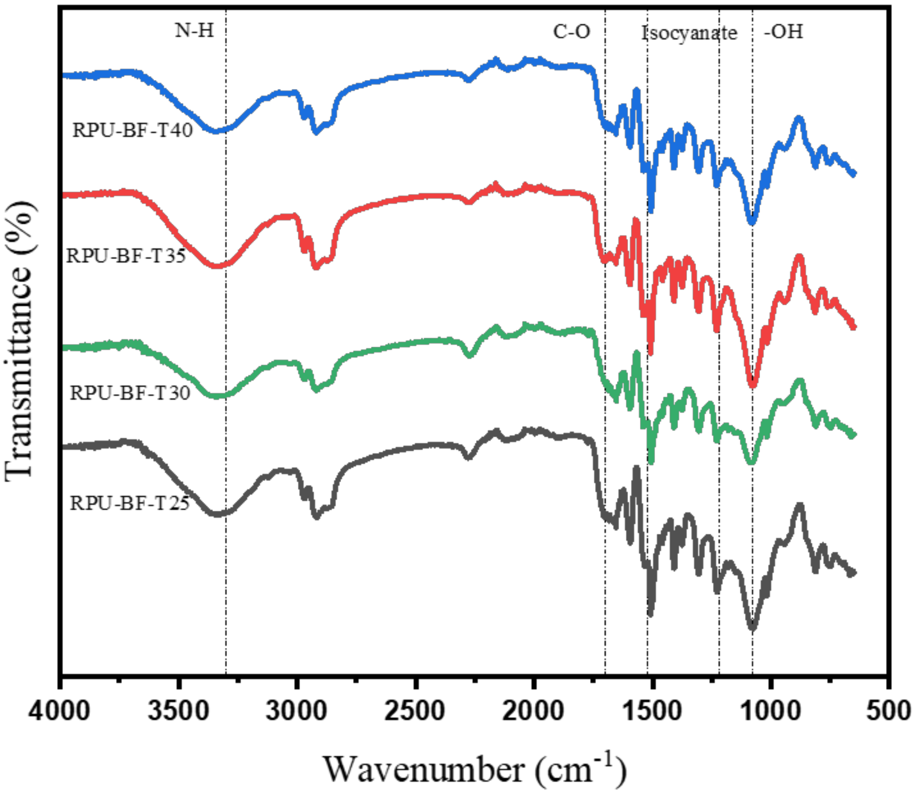


Fig. 9. FTIR spectrum of RPU-BF composite treated bamboo fiber

5. Conclusion

The purpose of this study was to determine the water contact angle of untreated and treated using silane coupling agent treatment for laminated bamboo fiber composite. From the results, it was concluded that the water contact of treated bamboo fiber was 192.5° and 160.2° for untreated fiber. The use of a silane coupling agent for bamboo fiber treatment indicated the presence of Si-O-Si and Si-O-C bonds based on FTIR analysis. The physical characteristics and morphological structure of RPU-BF composites containing untreated and treated bamboo fibers were successfully obtained. At the peak thermal degradation temperature of 350°C, treated bamboo fiber exhibited a weight loss of 50%, whereas the weight loss of untreated bamboo fiber was 80% at 320°C. The FTIR spectrum peak of treated bamboo fiber at 3010-3040 cm-1 was related to silane coupling agent bonding Si-O-Si are stronger indicating new chemical bonds have been created between bamboo fiber and silane coupling agent due to the ester bond from the cellulose, lignin, and hemicellulose was completely cleaved. Meanwhile, the FTIR spectra of the RPU-BF composite displayed almost similar functional groups present. The pore diameter size of bamboo fiber decreased after silane coupling agent treatment referred to RPU-BF-T30 composite which was possible in laminated roof applications.

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