



Enhancing *Schizostachyum brachycladum* bamboo fiber reinforced PLA composites via plant ash alkali treatment: Interfacial and mechanical performance for lightweight and protective engineering components

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ABSTRACT

Plant ash derived alkali treatment has been previously explored for bamboo fiber modification in cement-based composites. However, its effectiveness in polymer matrices, particularly hydrophobic poly(lactic acid) (PLA), remains largely unexplored. In this study, the novelty and environmentally friendly plant ash alkali treatment was applied to bamboo fibers to enhance interfacial adhesion in PLA-based biocomposites. Plant ash alkali treatment roughened fiber surfaces, improving wettability that enhanced the fiber matrix bonding. Untreated bamboo fiber reinforced PLA composites containing 5 wt%, 10 wt%, and 20 wt% fiber (BFPLA-5, BFPLA-10, BFPLA-20) and equivalent to plant ash-treated bamboo fiber composites reinforced PLA composites with same fiber loading with untreated fiber (APBFPLA-5, APBFPLA-10, APBFPLA-20) were fabricated via internal mixing followed by compression moulding to evaluate structure–property relationships. Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) confirmed partial removal of amorphous constituents and enhanced cellulose crystallinity after treatment, with APBFPLA-20 exhibiting the highest diffraction intensity at 22.2°. Scanning electron microscopy (SEM) revealed improved fiber–matrix interfacial contact and reduced void formation in treated composites. These interfacial improvements resulted in significant mechanical enhancement, where the tensile strength increased by approximately 170% compared with untreated BFPLA-20 (15.46 MPa), reaching about 73% of neat PLA strength, while the flexural strength achieved 96.07 MPa with increased hardness. Thermogravimetric analysis (TGA) showed improved thermal stability, with the degradation onset temperature increasing to 328.59 °C for APBFPLA-10, and dynamic mechanical analysis (DMA) confirmed enhanced viscoelastic performance. Overall, APBFPLA-10 demonstrated the most balanced mechanical and thermal performance, indicating that plant ash alkali treatment is an effective and sustainable interfacial engineering strategy for bamboo fiber reinforced PLA composites intended for lightweight structural and protective engineering applications.

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1. Introduction

Nowadays, the development of biodegradable polymers made from natural resources that are renewable, recyclable, reasonably priced, and ecologically acceptable has been substantially impacted by the growing environmental concern about plastic waste [1,2]. Poly (lactic acid) (PLA) have outstanding mechanical performance, nontoxicity, renewability, biodegradability, and biocompatibility and abundant make it highly in demand [3].

Traditional petroleum based such as polyethylene, polypropylene, and polystyrene were replaced by PLA as a promising alternative [4]. PLA is made by turning lactic acid into a polymer. The lactic acid comes from fermenting renewable crops like corn, wheat, rice, starch, and sugarcane [5]. PLA exhibits a higher tensile modulus and strength compare with common used petroleum—based polymers such as polyvinyl chloride (PVC), polypropylene (PP), polystyrene (PS) which makes it suitable for structural and engineering applications. In addition, its biodegradability and biocompatibility provide environmental and biomedical advantages, while its transparency enables applications in packaging and optical products [6–8]. However, the application of PLA is still hindered by its inherent brittleness, low heat distortion temperature, and relatively high cost [9]. Blending PLA with assorted polymers, particularly polyvinyl alcohol (PVA), functions as a considered strategy designed to improve its mechanical features and expand its versatility in applications [10]. The process improves the substance's capabilities and simultaneously advocates for environmental sustainability by including organic elements. In addition to this particular strategy, the limitations associated with PLA can typically be mitigated through the reinforcement of the material using natural fibers [3]. Natural fibers (NFs) offer numerous benefits, including their wide availability, renewability, non-toxicity, recyclability, superior mechanical performance, versatility, low density, reduced specific mass, and biodegradability. [11–16]. NFs also low cost compared to synthetic fiber which cost around 400 to 4000 USD per ton [17]. NFs have been used as reinforcements for PLA matrix to produce sustainable biocomposites because they are completely biodegradable, renewable, economically feasible, and widely available [17–20].

A study in India found that annual production of bamboo is approximately 4.6 million tons (Mt) [21]. Thus, proved that its ability to harvested in short range year due to the high yield and growth rate [11,14,22–24]. Selection of natural fibers like bamboo favourable than synthetics are due to less energy required [25]. Bamboo fibers (BFs), have the chemical composition of approximately 74% cellulose, 12% hemicellulose, 10% lignin, 3% moisture, and 0.4% wax, as shown in Fig. 1. Bamboo fibers used in composite materials provide significant environmental advantages, positioning them as a sustainable substitute for conventional fibers.

Moreover, bamboo fibers are biodegradable, breaking down naturally at the end of their lifecycle without leaving harmful environmental residues. Tensile test recorded high value with 167.87 MPa marked that the mechanical performance of unidirectional continuous bamboo fiber-reinforced epoxy composites [26,27].

Therefore, the investigation into the modification of bamboo fibers is of paramount importance for the enhancement of the performance of bamboo fiber-reinforced composite materials.

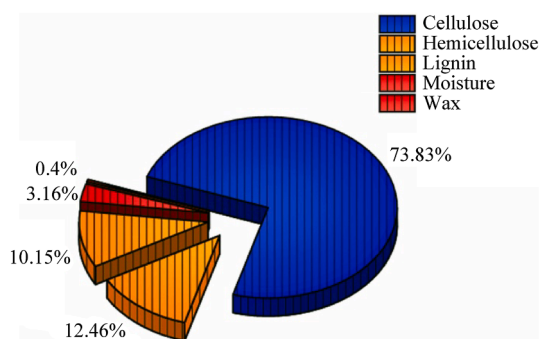


Fig. 1. Chemical composition of bamboo fiber [23].

Through surface modification or treatments would help diminish the polarity of bamboo fibers and help to augment the wettability and bonding strength between the fibers and the matrix and also enhancing the interfacial adhesion with the matrix through "mechanical interlocking" [11]. As seen in Fig. 2, common modification methods include alkaline treatment [28,29], silane coupling agent treatment [30], acetylation [31], benzoylation, permanganate treatment [32], isocyanate treatment [33] and maleic anhydride treatment [34].

Due to its effectiveness in removing surface impurities, reducing hydrophilicity, cost-effectiveness, high efficiency, and lack of organic pollutants, alkaline treatment has become one of the most popular methods in engineering applications [5,28,29,38,39]. Conventional sodium hydroxide (NaOH) treatment, commonly known as alkaline treatment, has been proven to modify fiber surfaces by removing lignin, hemicellulose, and waxy layers, exposing cellulose skeletons. This process not only enhances chemical compatibility with polymer matrices but also improves physical compatibility by increasing surface roughness and larger interaction area for stronger fiber–matrix bonding [36]. However, interfacial stability and overall composite performance may be harmed by excessive or extremely concentrated alkaline treatment, which can harm fiber structure, lower tensile strength, and produce excessively hydrophilic surfaces [36,40–42].

Recently, interest has grown in the use of environmentally friendly alkaline sources, including plant ash and rice husk ash, which are obtained from CaO, K₂O, and SiO₂-rich agricultural residues [43]. Plant ash, which is produced by the combustion of vegetation like straw, rice straw, and wood has been considered as a low-cost and mild alternative to conventional alkali agents. Its weak alkalinity and the less corrosive aqueous solution make it safer and more compatible with the environment compared to chemical alkalis of higher strength. Due to the rich content of alkaline minerals in plant ash, such as potassium, calcium, and magnesium oxides, it is considered an environmentally friendly treatment medium for natural fibers modification [42]. The treatment can be carried out through soaking, boiling, or mechanical agitations depending on the desired surface modification and type of fiber. Furthermore, plant ash-derived alkaline solution has been found not only to provide sufficient alkalinity for the activation of fiber surface but also to facilitate the mineral deposition that forms an interfacial layer, improving the fiber–matrix bonding. Recently, it has been reported that bamboo fibers treated with plant ash have shown remarkable reinforcing effects in ultra-high-performance concrete (UHPC) applications [26]. Nonetheless, investigations into the modification of bamboo fiber with plant ash are still scanty, while mechanistic action, interfacial behavior, and varying treatment concentration effects remain unveiled.

On the other hand, despite its shortcomings in sustainability and possible negative effects on long-term bonding, the majority of current research still uses alkali treatment by NaOH for natural fibers, this study aims to fill the knowledge gap by investigating properties of untreated bamboo fiber reinforced PLA composites containing 5 wt%, 10 wt%, and 20 wt% fiber (BFPLA-5, BFPLA-10, BFPLA-20) respectively and equivalent to plant ash-treated bamboo fiber composites reinforced PLA composites with same fiber loading to untreated fiber respectively (APBFPLA-5, APBFPLA-10, APBFPLA-20) as compared to pure PLA in terms of physical characteristics including chemical interaction by FTIR and XRD. Tensile, flexural, and hardness mechanical testing were also carried out. Along with these characterizations, TGA was carried out for thermal properties as important new information about the plant ash-treated and untreated bamboo fiber reinforced PLA composites. The morphological characteristics were also obtained

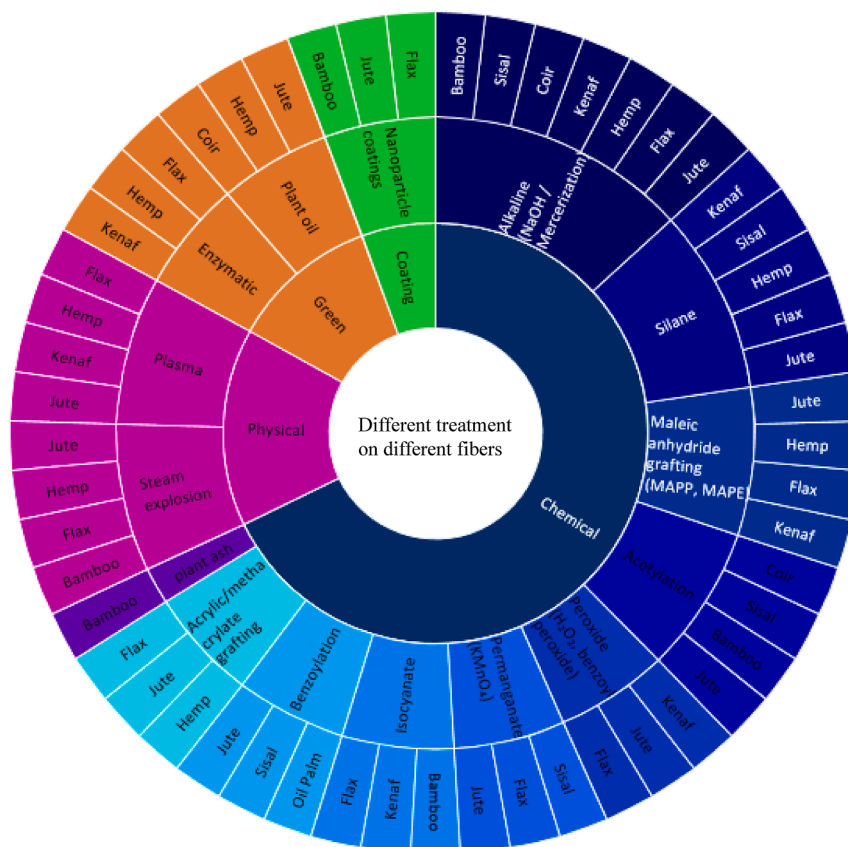


Fig. 2. Various type of fibers treatment [32,35–37].

using Atomic Force Microscopy (AFM) and SEM. The performance of sustainable PLA biocomposites reinforced with bamboo fiber will be more completely understood through to this study. Particularly for enhancing surface modification of natural fibers in biocomposites, this field of study has not been well investigated in earlier investigations. Additionally, the aim of this research is to offer a straightforward and affordable way for the weak characteristics of sustainable bamboo reinforced PLA biocomposites in which hinder the application of composites.

2. Experimental details

2.1. Materials

Mecha Solve Engineering, Malaysia provided Polylactic acid (PLA Nature Works USA, Ingeo 2003D, (CAS No. 26100-51-6)) with density 1.24 g/cm^3 , melt flow rate 6 g/10 min , and melting point $190 \text{ }^\circ\text{C}$ while Bamboo culm (*Schizostachyum brachycladum*) purchased from Kelantan and subsequently underwent a conversion process to obtain bamboo fibers, as illustrated in Fig. 3, Super Organic Enterprise supplied Plant Ash. Distilled Water from Spectrum Distillers Sdn Bhd.

2.2. Alkali treatment with plant ash

Bamboo fibers underwent plant ash alkali treatment as depicted in Fig. 4, using a 5%, 10%, 20% concentration by mass fraction. The solutions were agitated for 4 h at $80 \text{ }^\circ\text{C}$ and 2000 rpm on a magnetic stirrer plate. After the plant ash alkali treatment, bamboo fibers were cleaned with distilled water. Lignin, wax, fat,



Fig. 3. Process of making bamboo fibers.

hemicellulose, contaminants, and lower molecular weight substances were all eliminated by the wash. Finally, the treated fiber was dried for 24 h at $80 \text{ }^\circ\text{C}$ in an electric oven.

2.3. Fabrication of plant ash bamboo fiber reinforced PLA composites

As in Fig. 5, PLA and plant ash bamboo fiber reinforced PLA (APBFPLA) were melt blending through an internal mixer at a rotor speed of 50 rpm under $190 \text{ }^\circ\text{C}$ for 10 min. As shown in Table 1, the

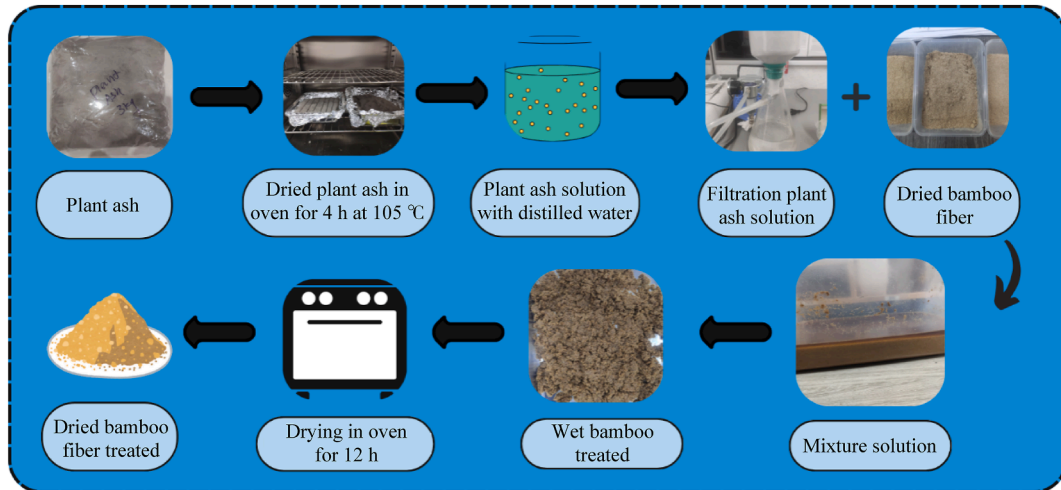


Fig. 4. Process of making plant ash bamboo fiber.



Fig. 5. Fabrication of plant ash bamboo reinforced PLA composites.

Table 1
Compositions of the composites samples.

| Samples | Weight/% | | |
|------------|----------|--------------|--------------------------------|
| | PLA | Bamboo fiber | Plant ash Bamboo fiber treated |
| PLA | 100 | 0 | 0 |
| BFPLA-5 | 95 | 5 | 0 |
| BFPLA-10 | 90 | 10 | 0 |
| BFPLA-20 | 80 | 20 | 0 |
| APBFPLA-5 | 95 | 0 | 5 |
| APBFPLA-10 | 90 | 0 | 10 |
| APBFPLA-20 | 80 | 0 | 20 |

ingredients were combined in various weight ratios. To create the composite sheets, the mixed samples were compressed using a 150 mm × 150 mm × 3 mm mold, 60 bar of pressure, a 190 °C

heating temperature, a 10-min preheat timer, a 13-min full press timer, a 3-vent cycle, and a 10-min cooling period with an 18 °C cooling temperature in a room temperature environment. Then samples cut into different size according to each characterization by bench saw. Same steps carried out again for untreated bamboo fiber reinforced PLA (BFPLA) composites.

2.4. Characterizations

2.4.1. Dynamic mechanical analysis

DMA Q800, Waters, TA Instruments, USA was utilized to examine each sample's glass transition temperature (T_g) and storage modulus. Strain ramping was the technique employed, and samples were 10 mm × 10 mm × 3 mm in size in a single-cantilever mode configuration in accordance with ASTM D5418-23 [44]. The temperature range was 30 °C to 150 °C, and the

frequency was 1 Hz.

2.4.2. Chemical composition

Invenio R, a Bruker model, was used to characterize FTIR spectra. The samples (10 mm × 10 mm × 3 mm) were scanned 32 times using the ATR diamond technique with a resolution of 4 cm⁻¹ at wavenumbers between 4000 and 400 cm⁻¹.

2.4.3. X-ray diffraction (XRD)

Rigaku SmartLab (2) and MiniFlex 600 (Rigaku Corporation, Japan) were used for XRD testing at 25 °C, 40 kV, and 30 mA. The 20 mm × 20 mm × 3 mm samples were scanned between 2θ = 5° and 60°.

2.4.4. Tensile test

The INSTRON 3366 Universal Testing Machine was used to test the composite samples for tensile properties in accordance with ASTM D638-14 [45], at room temperature with a crosshead speed of 2 mm/min were used for five repetitions per sample, each measuring 150 mm × 25 mm × 3 mm. All samples were measured for width and thickness using a digital caliper (CD-8C, Mitutoyo Corp, Japan) prior to testing.

2.4.5. Flexural test

In accordance with ASTM D790-17 [46], the composite samples were subjected to a three-point flexural test using the INSTRON 3366 Universal Testing Machine to determine flexural strength and modulus. A 10 kN load cell force at room temperature, a crosshead speed of 1 mm/min, a span length of 48 mm (16:1 ratio with sample thickness), and five readings per sample with dimensions of 127 mm × 13 mm × 3 mm were used in the testing. Prior to testing, a digital calliper (CD-8C, Mitutoyo Corp, Japan) was used to measure each sample's width and thickness.

2.4.6. Hardness test

Hardness test were conducted following the ASTM D2240-15 [47]. Test procedure was conducted utilizing a Shore D apparatus. Samples were cut out to dimensions of 10 mm (L) × 10 mm (W) × 3 mm (T) and five readings have been taken to get accurate results. The findings of this test will help to evaluate the material's hardness.

2.4.7. Atomic force microscopy (AFM)

The AFM (Dimension Edge with Scanasyt, Bruker Crest, USA) was used to examine the morphology of the composite samples. It had a height sensor data type, a 300-mV amplitude set point, a 5 μm scan size, and a 5 μm/s scan rate.

2.4.8. Scanning electron microscopy (SEM)

The sample's morphology was examined using an EM-30AX Plus (Coxem Co., Ltd., Korea) at 5 kV and 200 × magnification. The samples were coated with gold for 5 min using an SPT-20 coater (Coxem Co., Ltd., Korea) to lower the electron charge prior to observation.

2.4.9. Thermogravimetric analysis (TGA)

To assess weight loss as temperature rose, the thermal breakdown behaviour of the bamboo/PLA composites was examined using TGA. In this investigation, a TGA Mettler Toledo apparatus (Malaysia) was employed. The test was conducted at a flow rate of 40 mL/min in a nitrogen atmosphere. The temperature was gradually increased from 25 to 700 °C at a rate of 10 °C per min.

2.4.10. Statistical analysis

The experimental results of tensile, flexural and hardness

differences were statistically analyzed using IBM SPSS Statistics software. One-way analysis of variance (ANOVA) was employed to evaluate the significance of differences among the samples, followed by Tukey's honest significant difference (HSD) test for multiple comparisons at a confidence level of 95% ($p \leq 0.05$).

3. Results and discussion

3.1. Fourier transform infrared (FTIR)

The chemical structure of the untreated and ash-plant-treated bamboo fiber reinforced PLA biocomposites was analyzed by FTIR spectroscopy, as illustrated in Fig. 6. All spectra exhibit characteristic absorption bands of PLA, confirming that the incorporation of BF and APBF does not change the fundamental chemical structure of the polymer matrix. The noticeable peak located at approximately 1747–1749 cm⁻¹ corresponds to the C=O stretching vibration of the ester functional groups in PLA [48]. Peaks observed in the region of 1452–1360 cm⁻¹ are attributed to C–H bending, whereas the peak around 1180 cm⁻¹ is associated with C–O stretching within the ester linkage. The C–O–C asymmetric stretching band appears distinctly near 1080 cm⁻¹, and the peak found around 1042 cm⁻¹ indicates C–CH₃ vibrational modes, which are typical signatures of PLA molecular chains [49]. Additionally, the prominent peaks appearing at 868–866 cm⁻¹ and 755–752 cm⁻¹ correspond to the crystalline and amorphous regions in the polymer chain, respectively [50–52].

In contrast, BFPLA-20 presents a broad –OH absorption band centered at 3579 cm⁻¹, corresponding to the hydroxyl groups from lignocellulosic components of untreated bamboo fibers. The position at a lower wavenumber indicates that stronger hydrogen bonding interactions and the presence of bound hydroxyl groups take place, typical of untreated natural fibers with higher moisture affinity. Meanwhile, APBFPLA-20 exhibits a sharper absorption band at 3691 cm⁻¹, which is not attributable to free cellulose hydroxyl groups. In solid lignocellulosic materials, cellulose O–H stretching vibrations typically appear as a broad band in the 3000–3600 cm⁻¹ region due to extensive hydrogen bonding, whereas free hydroxyl groups above 3600 cm⁻¹ are rarely detected. Typically, free cellulose hydroxyls that appear above 3600 cm⁻¹ can be difficult to observe because of the way they interact with other elements in the lignocellulosic structure. The band at 3691 cm⁻¹ is therefore assigned to inorganic mineral hydroxyl groups, commonly associated with Ca²⁺ and Mg²⁺ containing phases such as Mg(OH)₂ or kaolinite-type structures introduced by the plant-derived ash treatment [53,54]. The high concentrations of alkaline-earth elements in the ash suggest that these minerals will be stored in the ash at the bamboo fibre

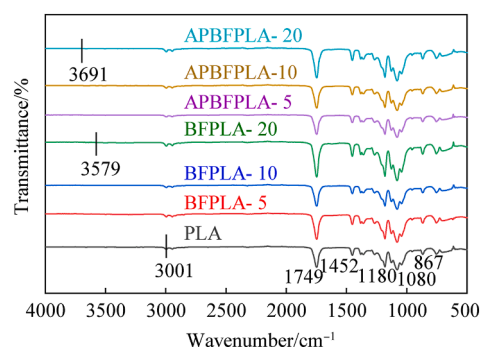


Fig. 6. FTIR spectra of plant ash treated bamboo and untreated bamboo fiber reinforced PLA composites.

surface. These minerals also create additional surface excitation and inhibit the formation of hemicellulose and lignin, contributing to increased roughness. Additionally, because they create barriers to thermally stable interfaces, improved mechanical properties and greater thermal stability within the composites containing ochre are consistent with those observed with the PLA composites containing the blended treatments [42].

Comparatively, the BFPLA and APBFPLA composites present similar peak positions to pure PLA. No significant peak shift or emergence of new functional groups is observed across all fiber loadings (5 wt%, 10 wt%, and 20 wt%). This condition suggests that both untreated and treated bamboo fibers do not chemically react with the PLA matrix to form new bonding structures but rather interact mainly through physical interlocking or secondary bonding. The primary difference lies in the peak intensity variation, particularly in the treated fiber composites, which indicates improved interfacial interactions and reduced presence of hydroxyl with rich lignocellulosic components exposed at the composite surface.

3.2. X-ray diffraction (XRD)

To further investigate the structural features Fig. 7, illustrate XRD patterns of pure PLA compared to BFPLA and APBFPLA biocomposites. The broad diffraction features observed below 20° (2θ), particularly at 9.34° and 15.7° , denote the amorphous nature of PLA in all samples [55,56]. This broad scattering indicates the semi-crystalline behaviour of PLA reflecting its inherently slow crystallization kinetics under standard processing conditions.

A crystalline peak that appears in all samples around 22.2° (2θ) corresponds to the (002) crystal plane and reflects crystalline regions of both PLA and cellulose-rich fibres [57]. In contrast, this peak has greater intensity and becomes sharper for both BFPLA and APBFPLA composites, which BFPLA-10 shown the greatest peak intensity and suggest crystallite formation at moderate loading. This show as evidence that treated bamboo fiber led to the formation of new hydrogen bonds between cellulose chains. BFPLA-20 shown peak intensity slightly decrease due to the non-uniform dispersion of bamboo fiber with polymer chain hindering crystallization.

In contrast, the APBFPLA-20 composites show greatly improved crystallinity. The peak at 22.2° is most pronounced in a composite containing 20% fibre loading, indicating that higher molecular ordering and lattice perfection were achieved. Such improvement can be attributed to green alkali treatment by removing amorphous components such as lignin and hemicellulose, increasing the cellulose content, and enhancing the fibre-matrix interactions [42]. The green alkali treatment favors hydrogen bonding

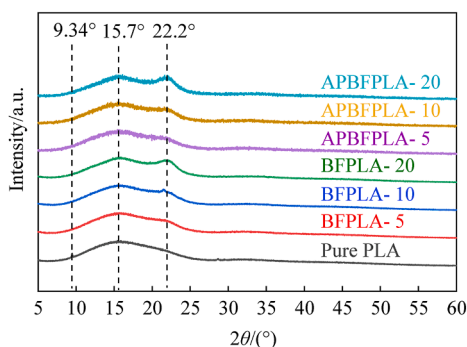


Fig. 7. XRD patterns of plant ash treated bamboo and untreated fibers reinforced PLA biocomposites.

interactions between PLA and cellulose, therefore allowing a tighter chain packing that increased crystallinity, which supports the findings reported recently.

These findings confirm that fiber content and surface treatment play an important role in influencing the crystallization behaviour of PLA. Higher crystallinity generally confers enhanced mechanical and thermal properties, thus making APBFPLA composites, especially at 20% fibre loading, highly suitable for applications that require structural rigidity, for example, biodegradable furniture, consumer products, and packaging materials.

3.3. Tensile strength

The tensile strength results of pure PLA, BFPLA, and APBFPLA composites are summarized in Fig. 8(a). Pure PLA recorded the highest tensile strength of 57.18 MPa, consistent with the high stiffness but brittle nature of neat PLA reported in previous studies [58]. Upon incorporation of untreated bamboo fibers, the tensile strength of BFPLA composites decreased progressively with increasing fiber loading, with BFPLA-5, BFPLA-10, and BFPLA-20 exhibiting 41.78 MPa, 31.38 MPa, and 15.46 MPa, respectively. The clear reduction of up to 73% at 20% fiber content is attributed to poor interfacial adhesion between the hydrophilic bamboo fibers and the hydrophobic PLA matrix, which reduces stress transfer efficiency and accelerates crack propagation [59,60].

In contrast, the plant ash-treated bamboo fiber reinforced PLA

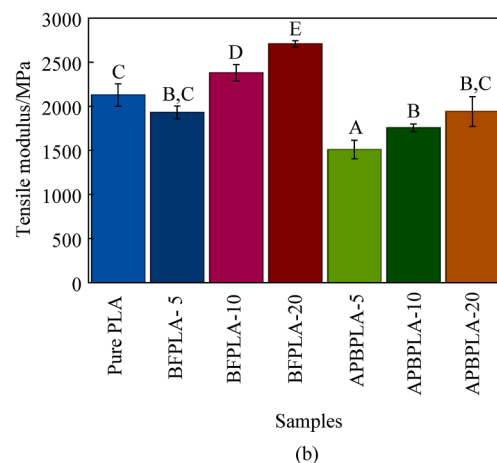
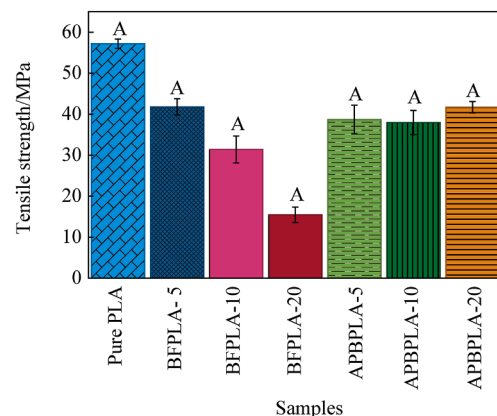


Fig. 8. (a) Tensile strength; (b) Tensile Modulus data of plant ash treated bamboo and untreated fibers reinforced PLA Composites.

Note: Values with different letters in the figures are significantly different ($p < 0.05$).

(APBFPLA) composites exhibited comparatively improved tensile performance. APBFPLA-5, APBFPLA-10, and APBFPLA-20 recorded tensile strengths of 38.69 MPa, 37.96 MPa, and 41.68 MPa, respectively, with APBFPLA-20 retaining nearly 73% of neat PLA's tensile strength. This improved performance suggests that plant ash treatment may enhance fiber matrix compatibility due to better removal of surface impurities and partial modification of hydroxyl groups, which promotes improved dispersion and enhanced interfacial bonding "mechanical interlocking" [3,36]. Although both BFPLA and APBFPLA composites exhibited lower tensile strength than neat PLA, the APBFPLA series consistently retained higher strength values across all loadings, highlighting the potential of plant ash treated bamboo fibers as a promising reinforcement alternative offering more stable mechanical performance in PLA-based biocomposites.

Fig. 8(b) shows the tensile modulus of the untreated and treated bamboo fibers reinforced PLA composites. Although tensile modulus of pure PLA was 2127.43 MPa, reflecting its inherently stiff but brittle nature. BFPLA composites demonstrated varying modulus responses, where BFPLA-10 achieved a moderate enhancement 2379.82 MPa, indicating increased stiffness at optimal fiber content compared to pure PLA that recorded 2127.43 MPa reflect its properties a brittle nature while excessive fiber addition with 20% loading (BFPLA-20) with value 2707.56 MPa resulted in brittleness and reduced toughness due to micro voids are mostly created at high fiber loading because of poor interfacial adhesion between the fiber and matrix, inadequate fiber wetting, and fiber-matrix debonding [61]. On the other hand, APBFPLA composites displayed competitive but more stable modulus values APBFPLA-5 (1509.12 MPa), APBFPLA-10 (1756.02 MPa), and APBFPLA-20 (1940.73 MPa) suggesting that plant ash treatment contributes to more uniform fiber dispersion and balanced stiffness. Table 2 summarizes the tensile strength and tensile modulus of the composite samples, highlighting the influence of fiber loading and plant ash alkali treatment. Overall, while BFPLA composites exhibited slightly higher modulus values, APBFPLA demonstrated superior consistency and interfacial stability, confirming that optimized natural fiber treatment can yield more balanced mechanical properties in PLA-based composites [62].

3.4. Flexural strength

Flexural strength is the maximum bending stress a material can withstand before fracture when subjected to a three-point bending test [63]. As presented in Fig. 9(a), neat PLA presented the highest flexural strength value of 74.74 MPa and was thus set as a benchmark. Reinforcement with untreated bamboo fibers (BFPLA series) significantly reduced the flexural strength: BFPLA-5, BFPLA-10, and BFPLA-20 yielded 63.73 MPa, 62.33 MPa, and 41.93 MPa, respectively, which approximately corresponds to a reduction of 14.7%, 16.6%, and 43.9% in comparison with pure PLA.

Table 2
Tensile strength and Modulus of the composites.

| Sample Code | Tensile Strength/MPa | Tensile Modulus/MPa |
|-------------|-------------------------|-------------------------------|
| Pure PLA | 57.18±1.27 ^A | 2127.43±141.48 ^C |
| BFPLA-5 | 41.78±2.24 ^A | 1930.68±82.29 ^{B,C} |
| BFPLA-10 | 31.38±3.67 ^A | 2379.82±103.89 ^D |
| BFPLA-20 | 15.46±2.12 ^A | 2707.56±41.64 ^E |
| APBFPLA-5 | 38.69±3.90 ^A | 1509.12±117.15 ^A |
| APBFPLA-10 | 37.96±3.29 ^A | 1756.02±47.88 ^B |
| APBFPLA-20 | 41.68±1.57 ^A | 1940.73±188.03 ^{B,C} |

Note: Values with different letters in the table are significantly different ($p < 0.05$).

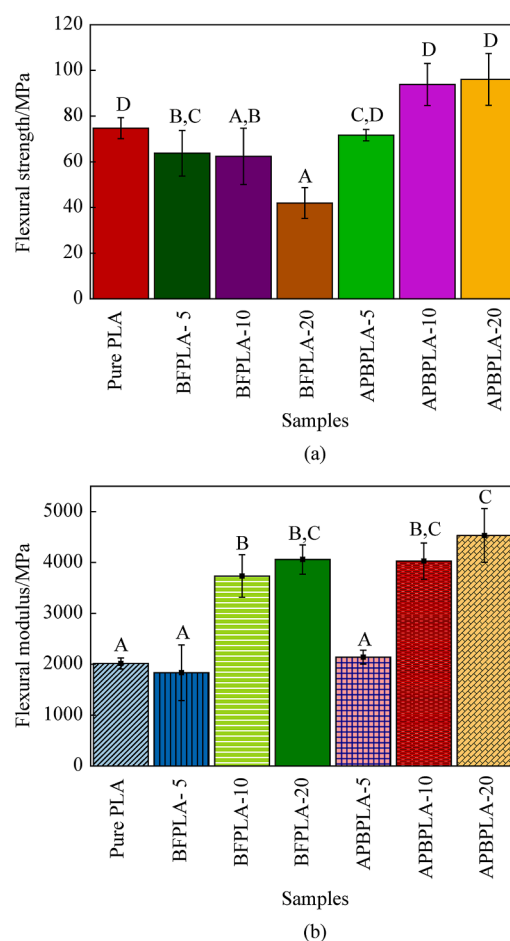


Fig. 9. (a) Flexural strength; (b) Flexural modulus data of plant ash treated bamboo and untreated fibers reinforced PLA composites.
Note: Values with different letters in the figures are significantly different ($p < 0.05$).

These findings result from poor matrix and fiber aggregation in the composite, which weakens the fiber/matrix link and reduces the composite's strength [64]. Compared to the tensile test, the flexural test frequently shows a progressive failure of the composite and a larger produced stress with a modest load. Flexural strengths were significantly greater than tensile strengths. Weibull's statistical strength theory states that because of the nature of stress fluctuation, some materials have higher bending strengths than tensile strengths. Flexural strengths were significantly greater than tensile strengths. Weibull's statistical strength theory states that because of the nature of stress fluctuation, some materials have higher bending strengths than tensile strengths. In contrast, the composite will break readily in a tensile test because the load is dispersed throughout its cross-section, which causes more faults to occur and even the weakest fiber to start a crack [61].

By contrast, the addition of plant ash treated bamboo fibers significantly improved the flexural strength compared with that of untreated fiber composites, APBFPLA-5 reached about 71.62 MPa, retaining 95.8% of neat PLA's strength; APBFPLA-10 and APBFPLA-20 showed excellent flexural strengths of 93.84 MPa and 96.07 MPa, respectively, which was an enhancement of 25.5% and 28.5%, respectively, compared to pure PLA. This great improvement suggests that the treatment of plant ash promoted fiber-matrix compatibility, leading to an effective stress transfer under the condition of a bending load.

Similarly, Fig. 9(b) showed that the flexural modulus had clear

stiffness changes with the incorporation of plant ash treated bamboo fiber. Pure PLA had a modulus of 2015.56 MPa, and BFPLA-5, when incorporating low amounts of fiber, had a decreased value of 1833.7 MPa due to weak bonding [65]. However, higher bamboo fiber contents increased the stiffness, with BFPLA-10 and BFPLA-20 at 3735.08 MPa and 4060.69 MPa, respectively [66]. APBFPLA composites further improved the stiffness properties, with APBFPLA-5 slightly above neat PLA 2139.06 MPa, APBFPLA-10 having a remarkable modulus of 4026.7 MPa, and APBFPLA-20 having a modulus of 4531.86 MPa. It is thus confirmed that both bamboo fiber and plant ash treatment are effective in reinforcing the PLA matrix to make the material more rigid and dimensionally stable. Improved mechanical performance of APBFPLA composites consequently points out their potential for high-strength applications that are eco-friendly in nature, such as sustainable packaging and furniture [3]. Table 3 summarize the flexural strength and flexural modulus of the composite samples.

3.5. Hardness test

The Shore D hardness of pure PLA was 76.5, as depicted in Fig. 10, consistent with literature-reported values around 75–80. Incorporating untreated bamboo fibers increased hardness (79.4–81.5), indicating that rigid fibers restrict PLA chain mobility and raise surface stiffness. At 5 wt% loading (81.1), good dispersion aided stress transfer, but at 10 wt% (79.4) slight agglomeration and weak interfacial bonding reduced hardness. With 20 wt% bamboo fiber, hardness recovered (81.5), suggesting that a higher number of rigid particles can still resist indentation even if bonding is imperfect. After ash-plant treatment, APBFPLA composites showed even higher hardness (81.1–84.0), peaking at APBFPLA-10 (84.0), likely due to improved interfacial bonding via removal of hemicellulose/lignin and increased surface roughness. However, at 20 wt% treated fiber (81.1), there was a slight decline, likely because excessive fiber content caused agglomeration and reduced bonding. Overall, treated fibers consistently outperformed untreated ones, and 10 wt% treated fiber loading provided the best improvement in PLA hardness [67]. Table 4 summarize the shore D hardness test of the composite samples.

3.6. Scanning electron microscope (SEM)

Fig. 11 depicts the macroscopic and SEM of tensile cross sectional fracture surfaces of the untreated and treated bamboo-fiber/PLA composites. Figs. 11(b)–11(g) display that fiber–matrix interactions dominate the mechanical behavior. The fractured surfaces of untreated BFPLA samples, shown in Figs. 11(b), 11(d), and 11(f), reveal distinctive evidence for weak interfacial bonding: widespread fiber pull-out, interfacial gaps, debonded fiber imprints, and voids in agreement with literature reports for poor adhesion between hydrophilic natural fibers and PLA [68,69]. BFPLA-5 evidences partial pulling out of fibers, which infers partial

Table 3
Flexural Strength and Modulus of the composites.

| Sample Code | Flexural strength/MPa | Flexural Modulus/MPa |
|-------------|----------------------------|-------------------------------|
| Pure PLA | 77.79±4.33 ^D | 1987.54±112.74 ^A |
| BFPLA-5 | 54.51±9.99 ^{B,C} | 1833.70±547.35 ^A |
| BFPLA-10 | 45.56±12.32 ^{A,B} | 3735.08±416.71 ^B |
| BFPLA-20 | 32.96±6.75 ^A | 4060.70±288.31 ^{B,C} |
| APBFPLA-5 | 69.24±2.49 ^{C,D} | 2139.06±135.89 ^A |
| APBFPLA-10 | 79.43±9.21 ^D | 4026.69±356.31 ^{B,C} |
| APBFPLA-20 | 84.96±11.35 ^D | 4531.86±527.67 ^C |

Note: Values with different letters in the table are significantly different ($p < 0.05$).

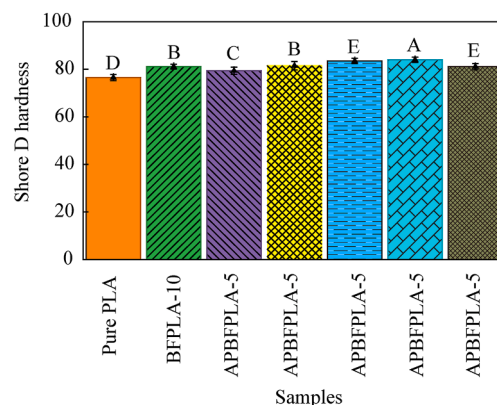


Fig. 10. Hardness data of plant ash treated bamboo and untreated fibers reinforced PLA composites.

Note: Values with different letters in the figures are significantly different ($p < 0.05$).

Table 4
Shore D hardness test of the composites.

| Sample Code | Shore D Hardness |
|-------------|-------------------------|
| Pure PLA | 76.80±0.30 ^D |
| BFPLA-5 | 81.13±0.15 ^B |
| BFPLA-10 | 79.73±0.31 ^C |
| BFPLA-20 | 81.13±0.32 ^B |
| APBFPLA-5 | 69.30±3.31 ^E |
| APBFPLA-10 | 83.93±0.21 ^A |
| APBFPLA-20 | 81.20±0.26 ^E |

Note: Values with different letters in the table are significantly different ($p < 0.05$).

stress transfer, while BFPLA-10 shows increased pull-out and matrix tearing. With further increases in BF loading to BFPLA-20, the agglomeration of fibers and the development of voids become significant, developing into stress concentrators that promote early crack nucleation, consistent with a reduction in strength.

By contrast, the treated APBFPLA composites (Figs. 11(c), 11(e), and 11(g)) exhibited significantly enhanced interfacial compatibility, consistent with reports of improved wetting and adhesion in PLA–natural fibre systems subjected to treatments [65,68]. APBFPLA-5 presents fewer voids and an improved fibre–matrix bonding, whereas for APBFPLA-10, the fibres are broken evenly with the matrix surface, and river-mark patterns can be seen, which evidences that the composite was transferring load effectively before it fractured. While some voids and clusters still exist in APBFPLA-20, a reduction in fibre pull-out with increased presence of fibre breakage confirms improved bonding compared with untreated BFPLA. This transition from pull-out-dominated failure to fibre-breakage behaviour directly relates to the higher flexural strengths demonstrated by APBFPLA samples. Remaining defects at high loadings indicate that processing and dispersion must still be optimized if the reinforcement potential is to be maximized.

3.7. Dynamic mechanical analysis (DMA)

Viscoelastic properties were measured by DMA. Fig. 12 presents the storage modulus (E') of PLA, BFPLA, and APBFPLA composites. PLA had the lowest storage modulus (E') of ~1500 MPa at room temperature, showing a sharp drop around 55–70 °C corresponding to its glass transition (T_g), indicating its brittle nature and thermal [70]. The presence of untreated bamboo fibers increased the modulus at lower temperatures due to the rigidity of the fibers

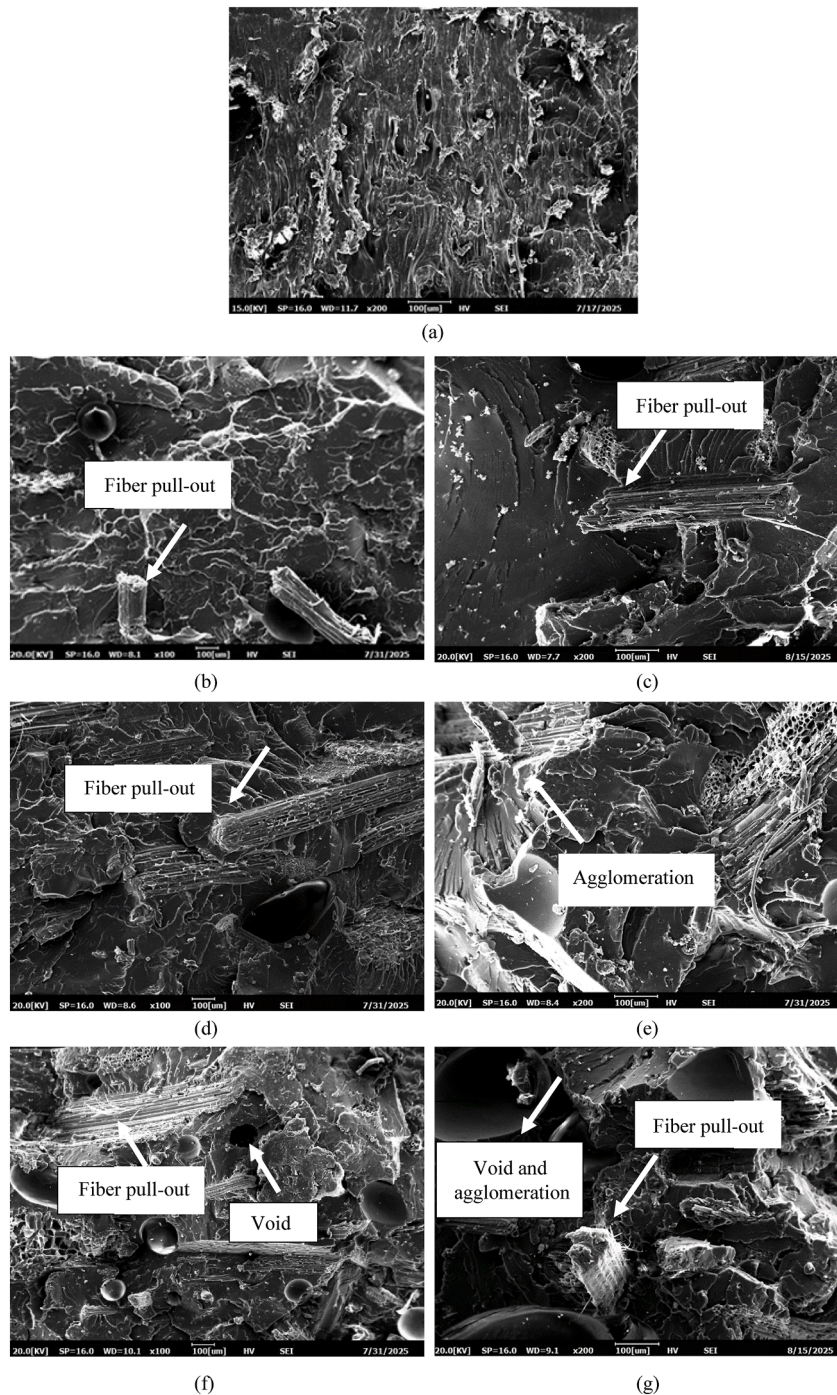


Fig. 11. SEM figures showing (a) Pure PLA; (b) BFPLA-5; (c) APBFPLA-5; (d) BFPLA-10; (e) APBFPLA-10; (f) BFPLA-20; (g) APBFPLA-20 composites.

[71]. Meanwhile at higher loadings, for example, BFPLA-20, rapid modulus decline beyond T_g is observed due to weak interfacial adhesion caused by surface lignin, waxes, and hemicellulose that restricts stress transfer [72].

In sharp contrast, the APBFPLA composites treated by plant ash treatment showed significant improvement in both stiffness and thermal stability. APBFPLA-5, APBFPLA-10, and APBFPLA-20 all possessed higher E' value compared to neat PLA and BFPLA, and APBFPLA-20 surpassed 2000 MPa in the glassy region. The improvement originates from the removal of amorphous components after treatment, as the cellulose hydroxyl groups are

exposed, the fiber-matrix adhesion is strengthened, the mobility of polymer chain is confined, and T_g is slightly elevated. Besides, the secondary increase in E' over 100 °C for APBFPLA-20 would be attributed to cold crystallization, which treated fibers worked as nucleation agents and hence lead to an improvement of PLA crystallinity [73,74].

Comparing treated and untreated composites at the same fiber loadings, it was observed that APBFPLA outperformed BFPLA consistently: APBFPLA-5 showed high stiffness over a broader temperature range, APBFPLA-10 maintained its modulus well beyond T_g , and APBFPLA-20 showed higher stiffness and a

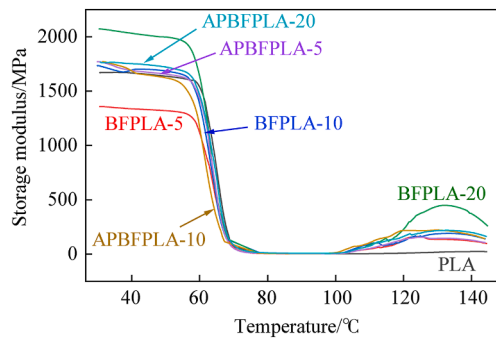


Fig. 12. DMA graph storage modulus of plant ash treated bamboo and untreated fibers reinforced PLA composites.

secondary rise of modulus, while BFPLA-20 softened prematurely. These confirm that fiber surface treatment indeed enhances the load transfer, viscoelastic behavior, and thermal-mechanical stability of the composite, particularly at higher fiber contents, in agreement with reports showing improved storage modulus by 20%–40% in treated natural fiber composites [72].

3.8. Atomic force microscope (AFM)

AFM analysis in Fig. 13 showed clearly different surface roughness of the composites between the untreated and the ash-treated bamboo fibers. In the case of the untreated BFPLA composites, the lowest roughness was at a moderate fiber loading, namely 20.9 nm for BFPLA-10, which indicated enhanced dispersion and partial encapsulation by the PLA matrix. The highest roughness occurred at low fiber loading, 68.9 nm for BFPLA-5, corresponding to poor interfacial adhesion and significant surface irregularities caused by the fiber pull-out effect. At high fiber loading, the roughness increased again for BFPLA-20, which had a roughness of 48.8 nm due to the agglomeration of fibers and disruption of matrix continuity [75].

In contrast, ash-treated composites (APBFPLA) displayed more uniform and moderate roughness values across all loadings, with the lowest roughness at 10 wt% fiber (36.3 nm) and the highest at 5 wt% (45.9 nm), slightly increasing at 20 wt% (44.2 nm). The reduced roughness compared to untreated composites indicates that chemical treatment enhanced interfacial adhesion by removing surface impurities and exposing cellulose hydroxyl groups, leading to smoother, more homogeneous surfaces and better stress transfer [75,76].

Comparison between the surface roughness of untreated and treated composites clearly illustrates the surface treatment's effect in reducing roughness, smoothing out irregularities, and promoting a more uniform morphology. While the presence of untreated fibers showed extreme roughness at low loadings and inconsistent trends with fiber content, treated fibers displayed balanced nanoscale topography regardless of loading, although optimal morphology was achieved at 10 wt%. These findings confirm that surface treatments improve fiber-matrix compatibility, thus enhancing the mechanical performance and reliability of PLA-based biocomposites [75].

3.9. Thermogravimetric analysis (TGA)

Fig. 14(a) illustrates TGA of PLA and its bamboo fiber composites. PLA recorded degraded at 333.57 °C, with minimal char residue due to nearly complete volatilization [77]. Incorporation of untreated bamboo fibers (BFPLA) lowered the maximum

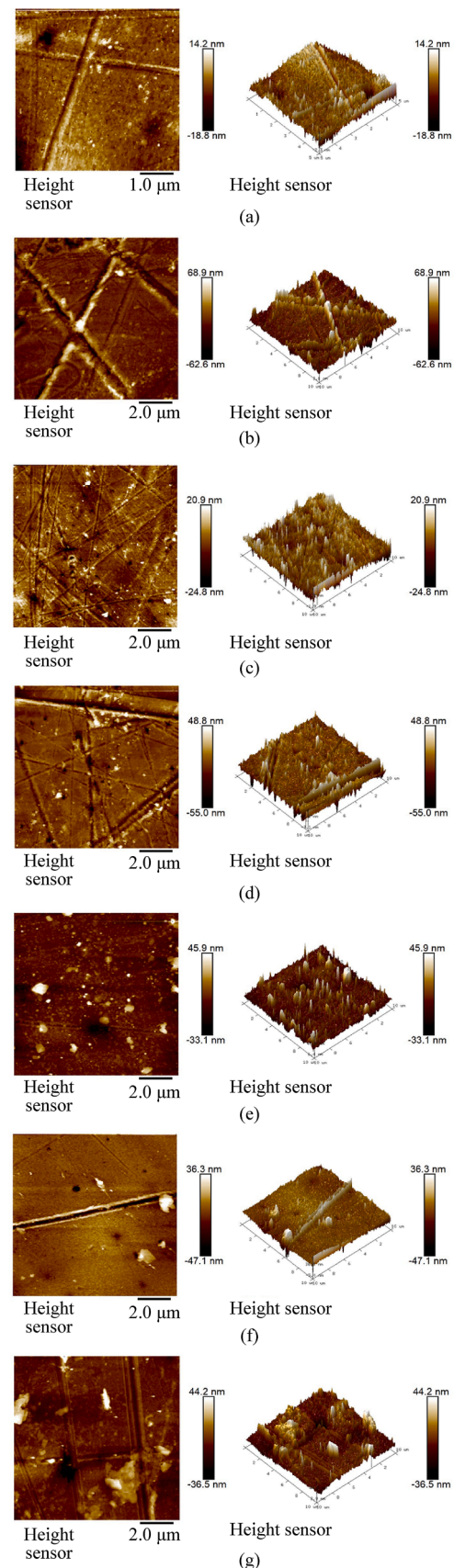


Fig. 13. Surface morphology through AFM; (a) Pure PLA; (b) BFPLA-5; (c) APBFPLA-5; (d) BFPLA-10; (e) APBFPLA-10; (f) BFPLA-20; (g) APBFPLA-20 composites.

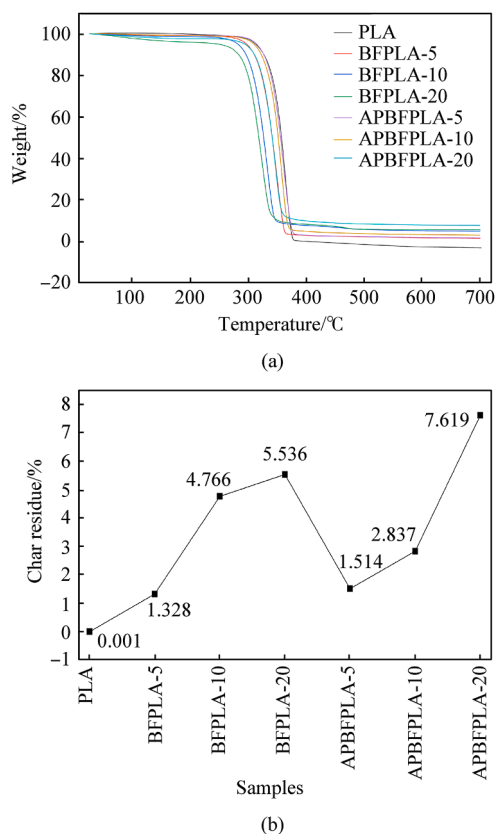


Fig. 14. (a) TGA thermogram of plant ash treated bamboo and untreated fibers reinforced PLA composites; (b) Residual weight fraction at 700 °C.

degradation temperature (T_{max}) from 310.16 °C (BFPLA-5) to 292.66 °C (BFPLA-20), reflecting the thermal instability of hemicellulose and lignin and poor fiber–matrix adhesion that accelerates decomposition. Char residue increased relative to PLA (Fig. 14(b)) but remained irregular due to weak interfacial bonding [78].

Ash-treated composites (APBFPLA) exhibited improved thermal stability, with T_{max} of 327.53 °C, 328.59 °C, and 311.32 °C for 5 wt%, 10 wt%, and 20 wt% fibers, respectively. This is due to plant ash may react with the removing lignin, hemicellulose, and waxes, exposing cellulose in the bamboo fibers, that could bring to enhance fiber–matrix adhesion and which delayed degradation may happened [79]. In addition, the inorganic mineral residues originating from plant ash, which are rich in Ca and Mg based compounds, act as a thermal barrier layer on the fiber surface, restricting heat transfer and volatile diffusion during thermal decomposition and further contributing to the increased thermal stability. Slight reduction in T_{max} at 20 wt% is attributed to fiber agglomeration and inhomogeneous dispersion, which locally accelerate decomposition.

Overall, treated composites showed higher char residue and more uniform thermal stability than untreated counterparts, with APBFPLA-10 achieving the optimal balance of T_{max} and char formation [79]. Excessive fiber loading, however, can reduce thermal performance due to agglomeration, highlighting the importance of optimizing filler content and distribution for PLA biocomposites. However, despite the significant improvements achieved through plant ash alkali treatment, further enhancement is still required for more sophisticated applications, as certain constraints remain. The focus of future studies may include incorporating other

nanoscale reinforcement materials, such as carbon nanotubes, graphene nanoplatelets, or nanoclays, to create a synergistic effect on the tensile properties of composite systems, while simultaneously improving thermal stability and enhancing durability under dynamic loading conditions. The enhanced mechanical strength and thermal stability suggest that these materials are suitable for non-structural and semi-structural engineering applications.

4. Conclusions

This study demonstrates that plant ash treated bamboo fiber (APBF) reinforced polylactic acid (PLA) composites exhibit markedly improved interfacial, mechanical, and thermal properties compared with untreated bamboo fiber reinforced PLA composites. The plant ash treatment effectively modified the fiber surface, leading to enhanced fiber matrix interaction and improved load transfer within the composite system. Among the investigated formulations, APBFPLA-10 exhibited the most balanced overall performance, achieving the highest flexural strength (96.07 MPa), maximum hardness, and the highest thermal degradation onset temperature (328.59 °C), while APBFPLA-20 showed the highest crystallinity peak (22.2°) and notable tensile strength enhancement approaching that of neat PLA. These results indicate that an intermediate fiber loading combined with plant ash treatment provides the most effective reinforcement strategy for PLA based composites. The findings confirm that plant ash treatment is an effective, environmentally benign surface modification approach for improving the performance of bamboo fiber reinforced PLA composites. The results demonstrate that plant ash-treated bamboo fiber reinforced PLA composites improved mechanical and thermal properties compared to untreated composites.

CRediT authorship contribution statement

M. Hazim Hidzer: Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis. **Azrol Jailani:** Writing – review & editing, Writing – original draft. **Vasi Uddin Siddiqui:** Writing – review & editing, Formal analysis. **S.M. Sapuan:** Writing – review & editing, Funding acquisition, Conceptualization. **E.S. Zainudin:** Writing – review & editing, Formal analysis. **A. Atiqah:** Supervision, Formal analysis.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: SM Sapuan reports financial support was provided by Putra Malaysia University. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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