

Thermal and mechanical evaluation of untreated kenaf fiber-reinforced nylon for FDM 3D-printed dental prostheses

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Abstract

Fused Deposition Modeling (FDM) 3D printing is utilized in dental appliances and prosthesis construction, eliminating the need for the complex laboratory procedures. One major downside of this technology is the poor mechanical properties. This study aimed to evaluate the reinforcement of nylon filament with untreated kenaf fibers for FDM 3D printing in terms of mechanical properties. Raw kenaf fibers were submitted to water retting, dried, ground, and sieved. The resulted fibers were combined with nylon beads at a concentration of 1 wt%. The modified nylon mix was used to produce filament by thermal compounding technique. The modified filament was used to print the experimental group's specimens while the as-received nylon filament was used to print the control specimens. Each group consisted of 5 specimens assigned for each testing method. Thermogravimetric analysis and differential scanning calorimetry was performed to the study samples. The mechanical evaluation was based on compressive strength, flexural strength, and surface hardness tests. The results were analyzed for significant difference by IBM SPSS software. Differential scanning calorimetry displayed optimized thermal stability after reinforcement. Although the mean value for compressive strength for the fiber-modified group was higher than the control group (49.34 ± 4.02 MPa and 45.02 ± 5.13 MPa, respectively), no statistically significant difference was observed between the groups (p -value $> .05$). However, there was a significant decrease in flexural strength for the fiber-modified group compared with the control group (23.23 ± 12.65 MPa and 43.14 ± 3.02 MPa, respectively) and p -value $< .05$. Surface hardness results showed no significant difference between the groups mean values (106.1 ± 1.14 for the control group, and 105.4 ± 1.85 for the fiber-reinforced group) with p -value $> .05$. The findings suggest that untreated kenaf fiber-reinforced composite at 1 wt% could provide acceptable mechanical behavior in applications require compression strength and scratching resistance. However, it may not tolerate bending stresses as expected.

Keywords

kenaf fibers, nylon, filament, FDM printing, fiber-reinforced composites

Introduction

3D printing is an additive manufacturing technology that creates physical objects based on digitally designed shapes. There are several types of 3D printing machines classified based on the printing method such as Stereo-lithography (SLA), Digital Light Projection (DLP), Selective Laser Sintering (SLS), PolyJet printing, and Fused Deposition Modeling (FDM) technologies. Fused deposition modeling (FDM) is a 3D printing technology that is based on polymer extrusion. It is considered to be simple and safe to operate compared with other 3D printing technologies. The printing materials used for this technology are thermoplastic polymers which are available in filament form. The printing process is based on liquefying and solidifying the filaments into shapes according to predesigned CAD models. There

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Data Availability Statement included at the end of the article

are several types of filament materials available for FDM printing technology such as PLA, ABS, Nylon, PETG, PEEK, and several modifications of these polymers as well.^{1–4} 3D printing technology has been utilized in medical and dental applications due to their ability to construct fine and detailed restorations with the exclusion of the complex laboratory procedures required for the conventional methods. Beside their precision, low cost, and easy handling by the practitioner, the work is digitalized and can be modified repeatedly.⁵ Nylon (polyamide) thermoplastic polymer is a suitable material for SLA and FDM-printed removable dentures. It possesses suitable mechanical properties which make it appropriate for short term usage.^{6,7} One of the significant downsides of FDM printing is poor mechanical properties, such as dimensional distortion with time, and weak flexural strength, mainly attributed to the buildup strategy involved in this technology.^{8,9} To overcome this issue, some polymers have been reinforced with fibers to optimize their mechanical strength and physical properties.^{10,11} Because of their low cost, availability, and their lower impact on the environment, natural fibers are considered for reinforcement rather than synthetic fibers.¹² Regarding FDM filament materials, there is limited literature focuses on the application of natural fibers reinforcement and their effect on the 3D-printed outcomes.

Kenaf plant, which is scientifically known as *Hibiscus cannabinus*, is an annual tall plant that can grow for up to 5 m within 4 months.¹³ Their stems are rich in fibers that can be extracted and modified to be used for a variety of purposes.¹⁴ The plant mostly exists in China, India, and some other parts of Asia while China and India represent 70% of the world kenaf fiber production.¹⁵ According to some literature, it was found that kenaf fibers can be used for reinforcement of some synthetic materials to enhance their mechanical properties.^{16–18} Due to the high significance and biosafety of this plant fiber, it has been suggested for reinforcing polymers and composites for biomedical applications. Natural fibers require special treatments prior to reinforcement with polymers to eliminate hemicellulose, lignin, and other unnecessary components. However, it has been pointed out by some literature that such treatment could possibly affect the mechanical strength of the fibers.^{19–21} Moreover, a recent study reported that alkaline-treated kenaf fiber-reinforced epoxy composite showed decreased mechanical strength compared with untreated kenaf fiber-reinforced composite. This finding was attributed to possible fiber pullout from the composite matrix and void formation.¹⁹ Our previous study investigated the mechanical and morphological behavior of alkaline-treated kenaf fiber-reinforced nylon for FDM 3D printing.²² The reduced mechanical strength of the fiber-modified nylon especially at 1 wt% concentration was found to be attributed to the reduced fiber strength as a result of alkaline treatment and poor fibers adhesion to the polymer matrix. Moreover, this exact concentration showed the least morphological

outcome compared with the lower studied concentrations. Building on this context, the present study aims to evaluate the effects of incorporating untreated kenaf fibers into a Nylon 6 (polyamide) matrix on the thermal and mechanical properties of the resulting composites fabricated via FDM 3D printing. This work seeks to provide insights into how untreated kenaf fibers influence the performance and suitability of FDM-printed nylon composites, addressing gaps in current knowledge regarding fiber treatment impacts and additive manufacturing of natural fiber-reinforced polymers.

Methods

Materials

Nylon filament (polyamide) Torewell™, China, was selected for this study. Polyamide pellets (nylon 6) were used for experimental specimens' preparation (QIPLAS, China). Grade A kenaf fibers (SKM2-Bio Grade A) from the National Kenaf and Tobacco Board, Kota Bharu, Kelantan, Malaysia, were used in this study for nylon reinforcement.

Preparation of fiber-reinforced filament

The kenaf fibers were initially subjected to a water retting process, which involved soaking the fibers in water for a duration of 24 h. This soaking facilitates the breakdown and removal of non-cellulosic materials, allowing for easier separation of the fibers. After the soaking period, the fibers were thoroughly washed to eliminate residual impurities and then left to dry naturally at room temperature for 48 h to ensure complete moisture removal. Once dried, the fibers were mechanically ground and subsequently passed through a sieve with a mesh size of 40 to obtain a uniform particle size suitable for composite production. These prepared fibers were then mixed with nylon beads at a weight concentration of 1% to produce the fiber-reinforced filament. The composite filament was fabricated using a thermal polymer extruder set at a temperature of 250°C, with the extruder single-screw rotating at a speed of 50 revolutions per minute to ensure homogeneous melting and mixing of the fiber and nylon components.

Specimens' preparation

The KF-reinforced filament was used to print the experimental group specimens, while the as-received nylon filament was used to print the control specimens. Specimens were printed using Ender 3 Neo 3D printer Creality, China. The printing speed was set to 100% (50 mm/s), and the infill was 100%. The nozzle temperature was 250°C and the bed temperature was 90°C. The specimen's dimensions were determined according to ASTM D695 and ASTM D790 for the mechanical testing while the dimensions were adapted within a permissible range as shown in Figure 1. Each group

consisted of 5 specimens for each test. Although this sample size is relatively small, it aligns with precedent in similar composite materials studies where resource and fabrication constraints limit specimen numbers.^{23,24}

Mechanical testing

Compressive strength, 3-point flexural strength tests were conducted using universal testing machine (Tinius Olsen, Model H50 KT, UK) as shown in Figure 2. The load cell was 50 KN and the test speed was 1 mm/min. The ultimate strength values were determined for each specimen,

representing the maximum load that each specimen could withstand prior to fracture or failure. Surface hardness was evaluated in this study using a Shore A durometer. For each rectangular specimen, three measurements were taken—at both sides and the center of the sample. The average of these three values was then calculated to represent the hardness of each individual specimen. The mechanical properties were compared between groups using IBM Statistical Package for Social Sciences (SPSS) version 20 independent t-tests. Alongside p-values, 95% confidence intervals (CI) for mean differences were calculated to provide an estimate of the precision and reliability of the observed effects.

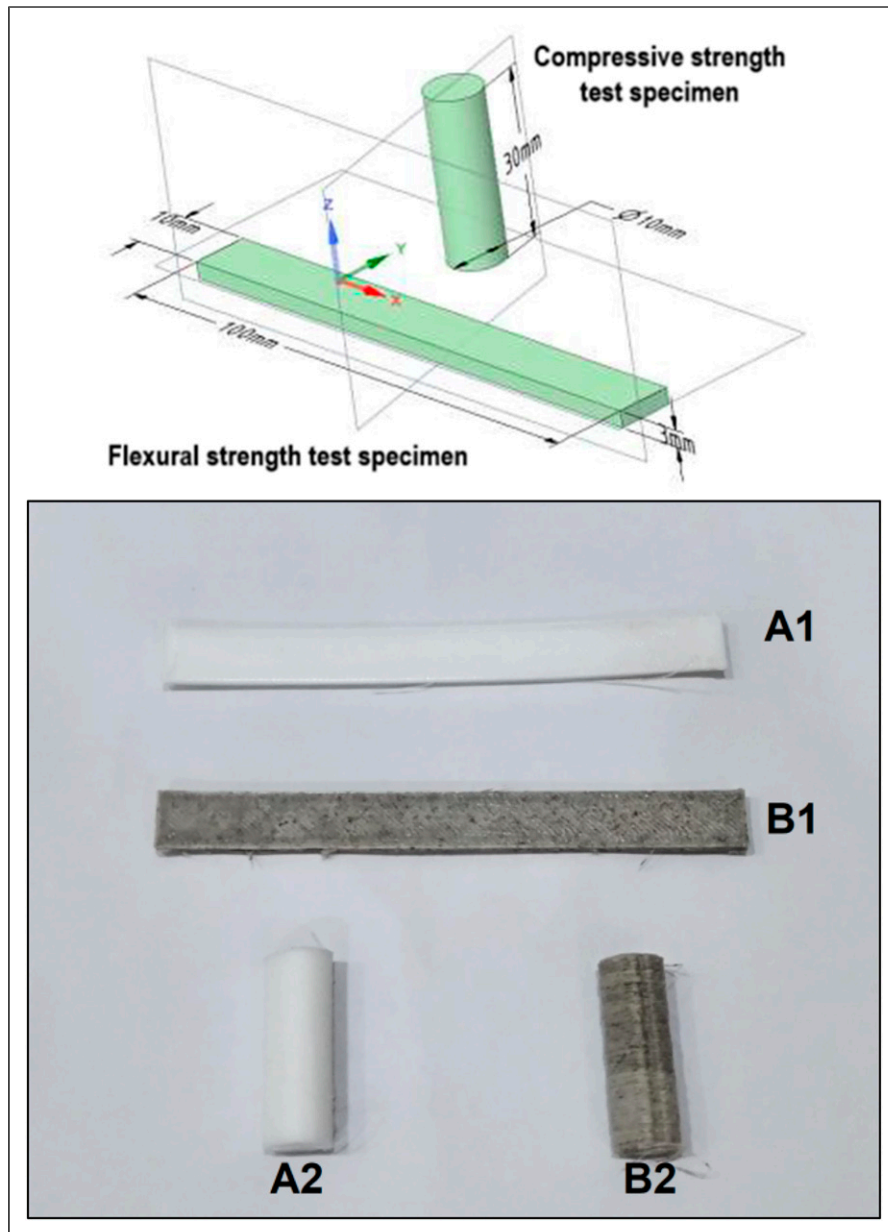


Figure 1. Specimens dimensions and form (A1 and A2:control specimen, B1 and B2 KF-reinforced specimen).

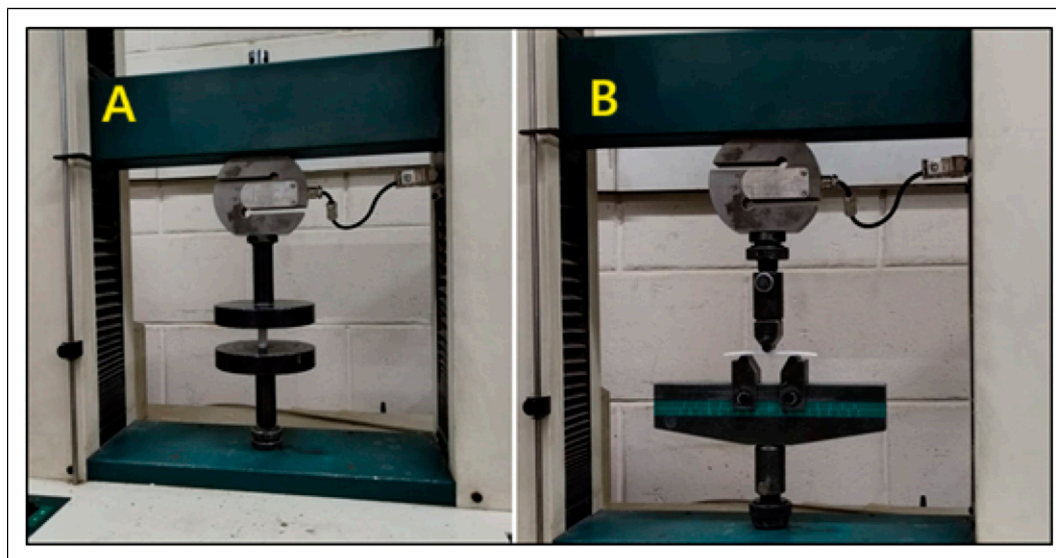


Figure 2. Universal testing machine (a) compressive strength test and (b) 3-point flexural strength test.

Thermal analysis

Thermogravimetric (TGA) analysis along with differential scanning calorimetry (DSC) tests (Shimadzu, Japan) were performed for the study materials to observe thermal behavior and stability after kenaf fibers reinforcement. TGA was conducted by taking a sample weight of approximately (5–10 mg) and placed in the platinum pan. The test was carried out under a nitrogen atmosphere with a flow rate of 50 ml/min. The samples were heated from room temperature to 600°C at a constant heating of 10°C/min.

Results

Thermal analysis

The thermal behavior of the nylon sample was analyzed using simultaneous DSC-TGA. The DSC curve showed a distinct endothermic melting peak within the range of 207.20°C to 226.18°C, with a peak melting temperature at approximately 226.18°C. The melting enthalpy was calculated to be 28.04 J/g, indicating a semi-crystalline structure typical of nylon. On the other hand, the TGA curve demonstrated that the material remained thermally stable up to around 400°C, after which a rapid degradation occurred. The maximum degradation rate was observed between 401.40°C and 463.83°C, resulting in a total weight loss of approximately 96.22%. These results confirm the high thermal stability of the nylon matrix prior to decomposition and support its suitability for processing and application in elevated temperature environments as in Figure 3.

The DSC-TGA analysis of the fiber-reinforced nylon composite revealed a melting temperature range between 218.01°C and 224.58°C, with a melting enthalpy of 6.291 J/

g, indicating reduced crystallinity compared to the neat nylon. Thermogravimetric analysis showed that thermal degradation began at approximately 489.02°C and peaked at 492.94°C. The total weight loss recorded was 96.25%, suggesting the presence of thermally stable fiber residues. These results demonstrate that the addition of kenaf fibers affected the thermal properties of the composite by reducing its crystallinity and slightly enhancing its thermal stability as in Figure 4. More comparable illustration is presented in Figures 5 and 6.

Mechanical testing

The mechanical properties of the Control and KF-reinforced groups were evaluated in terms of compression strength, flexural strength, and surface hardness. The KF-reinforced group demonstrated a higher mean compression strength (49.34 ± 4.02 MPa) compared to the Control group (45.02 ± 5.13 MPa), although this difference was not statistically significant (95% CI = $[-11.04, 2.40]$; $p = .177$). Conversely, a significant reduction in mean flexural strength was observed in the KF-reinforced group (23.23 ± 12.65 MPa) relative to the Control (43.14 ± 3.02 MPa) with a 95% CI (6.49, 33.32), and a p -value of 0.009, indicating a statistically meaningful difference. Surface hardness values were similar between both groups, with the control showing a mean of 106.1 ± 1.14 and the KF-reinforced group 105.4 ± 1.85 , without a statistically significant difference (95% CI $[-1.54, 2.94]$; $p = .492$) as illustrated in Figures 7–9.

Discussion

The thermal behavior of the control nylon, as revealed by DSC-TGA analysis, indicates a melting temperature in the

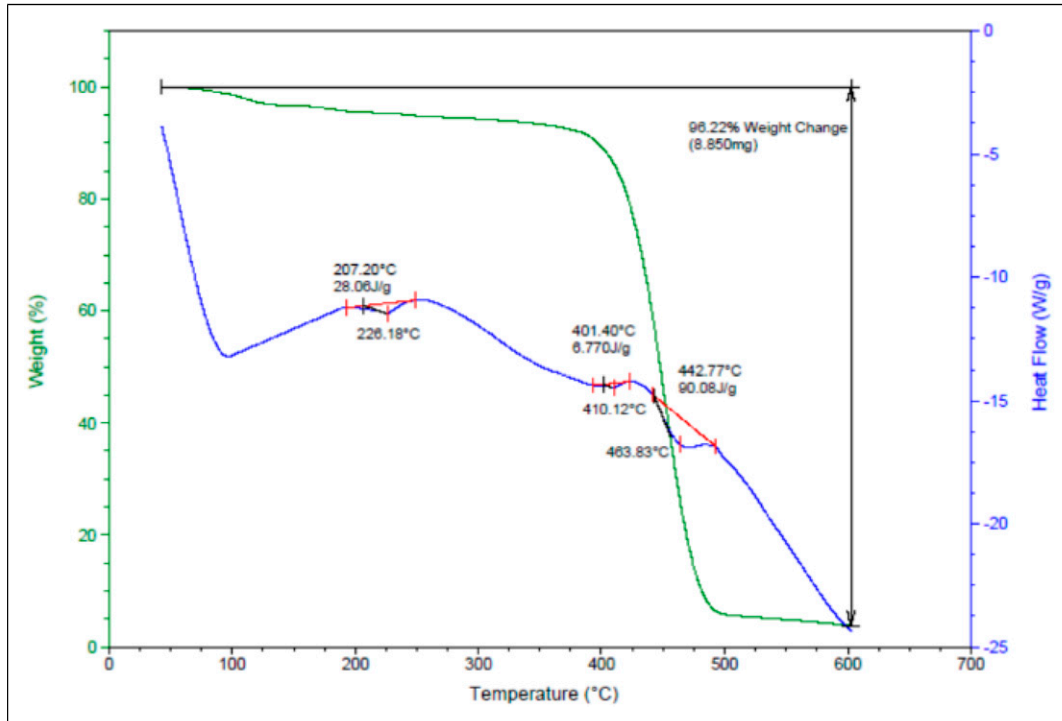


Figure 3. TGA and DSC for control sample.

range of 207.20°C to 226.18°C and a melting enthalpy of 28.06 J/g. This is consistent with the expected range for semi-crystalline nylon, suggesting that the base material retains its thermal integrity and processing ability under

standard conditions. Furthermore, the TGA results demonstrate that the nylon remains thermally stable up to approximately 400°C, with significant degradation occurring between 401.40°C and 463.83°C. The total weight loss of

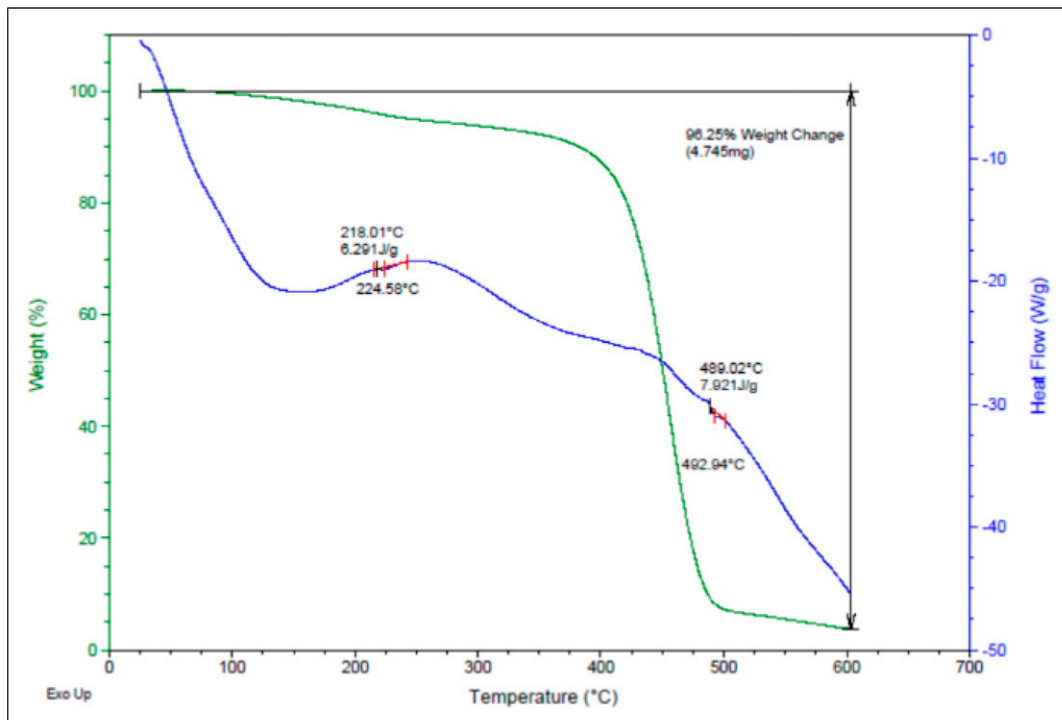


Figure 4. TGA and DSC for KF-reinforced sample.

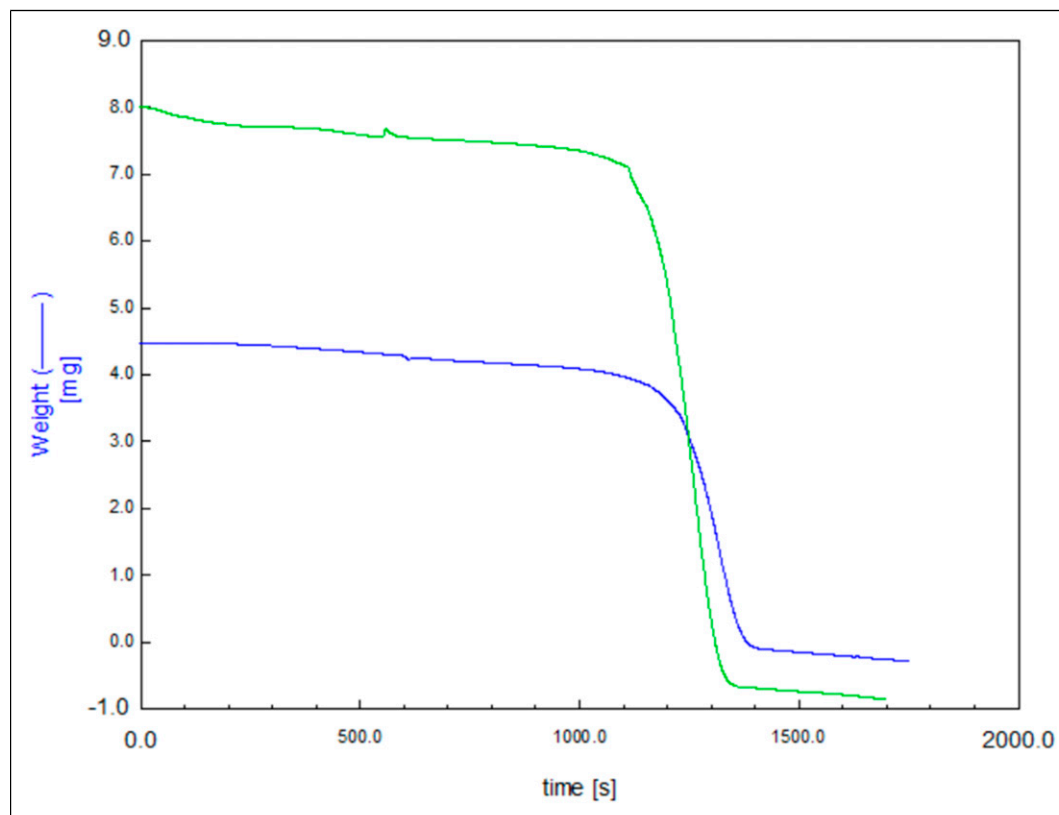


Figure 5. TGA (green = control, blue = KF-reinforced).

96.22% confirms complete decomposition at elevated temperatures.

The thermal profile of the KF-reinforced nylon composite exhibited noticeable differences compared to the neat nylon. The DSC analysis showed a slightly elevated melting point (224.58°C), yet a significantly reduced melting enthalpy (6.23 J/g), indicating a decline in the crystallinity of the polymer due to the presence of kenaf fibers. The reduction in melting enthalpy observed in the KF-reinforced composite is significant because it indicates a change in the composite's crystalline structure and thermal behavior. Melting enthalpy, measured by differential scanning calorimetry (DSC), reflects the amount of energy required to melt the crystalline regions of the polymer matrix. A decrease in melting enthalpy suggests a reduction in the degree of crystallinity or disruption of the crystalline phase by the incorporated kenaf fibers. The TGA curve revealed an onset of thermal degradation at approximately 489.0°C, similar to that of the control nylon. However, the maximum degradation temperature was extended to 492.94°C, indicating enhanced thermal stability, likely due to the thermal resistance of the fiber content. The total weight loss was approximately 96.25%, indicating the presence of thermally stable residues. These findings suggest that kenaf fibers influence both the thermal degradation pathway and crystallinity of the composite, which may impact processing

conditions and end-use thermal performance. The study results agree with a study findings conducted by Izzati and associates, that kenaf core fibers reinforcement to unsaturated polyester composite would significantly optimize the thermal behavior of the composite in terms of thermal stability.²⁵

The Mechanical results indicate that the KF-reinforced group exhibits an increased compression strength compared to the control, although this difference was not statistically significant that could be due to higher variability in measurements for the control group. This enhancement in compressive performance may be attributed to the reinforcing effect of the KF material, which likely improves load-bearing capacity under compressive stresses. However, the significant reduction observed in flexural strength for the reinforced group suggests that the incorporation of KF adversely affects the material's ability to withstand bending forces, potentially due to reduced ductility or increased brittleness introduced by the reinforcement. Surface hardness remained statistically unchanged, indicating that the KF reinforcement does not significantly alter the material's surface resistance to deformation or wear. The reduction in the composite crystallinity may relate to the observed mechanical properties by influencing the composite's stiffness, strength, and thermal stability. Lower crystallinity typically leads to decreased rigidity and strength, which

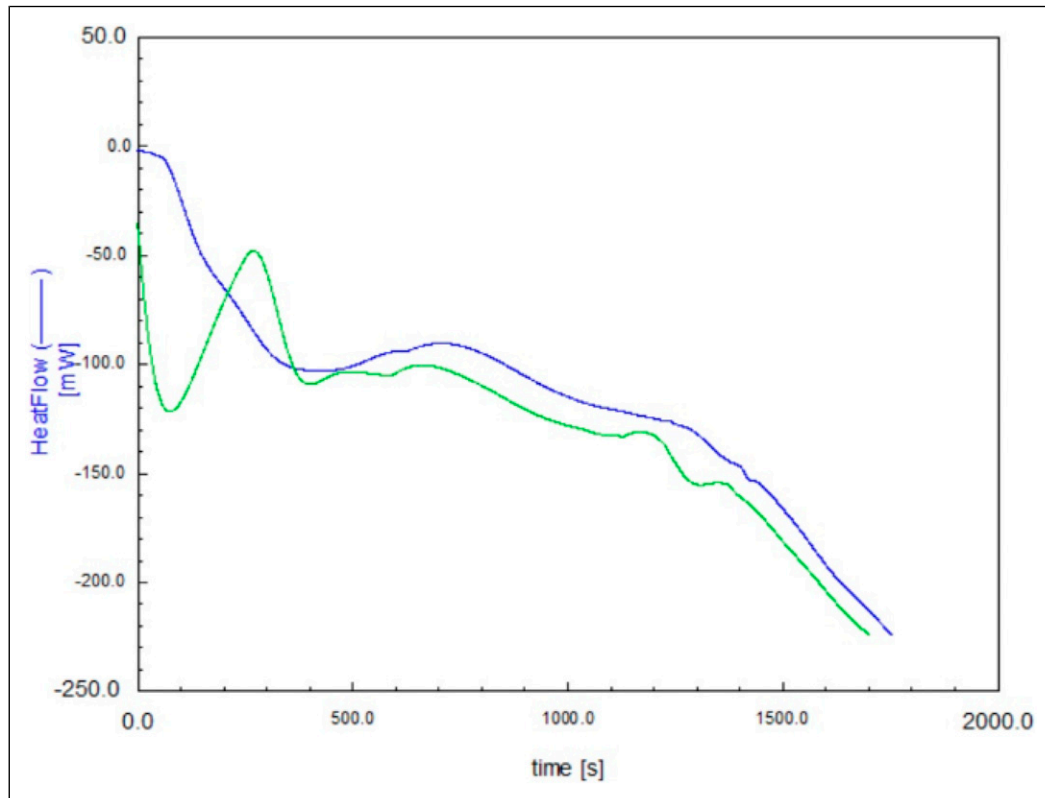


Figure 6. DSC (green = control, blue = KF-reinforced).

could explain reductions in flexural strength seen in the KF-reinforced composite. However, the fibers may simultaneously enhance other properties, such as surface hardness, by reinforcing the polymer matrix at the microstructural

level. In comparison with our previous work on alkaline-treated kenaf fiber-reinforced nylon composites,²² the present study employing untreated kenaf fibers exhibited a slightly higher compressive strength, while the flexural

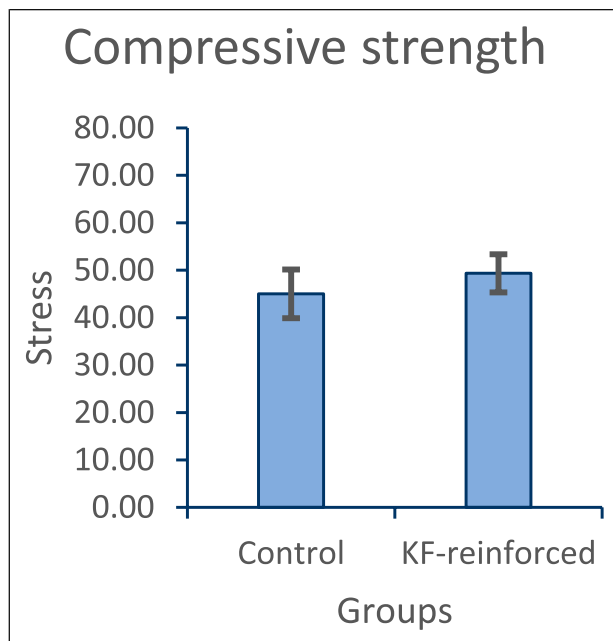


Figure 7. Compressive strength mean values.

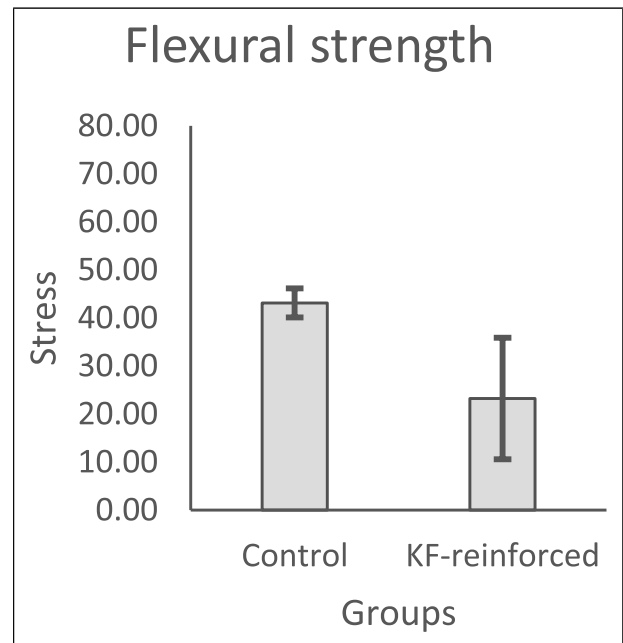


Figure 8. Flexural strength mean value.

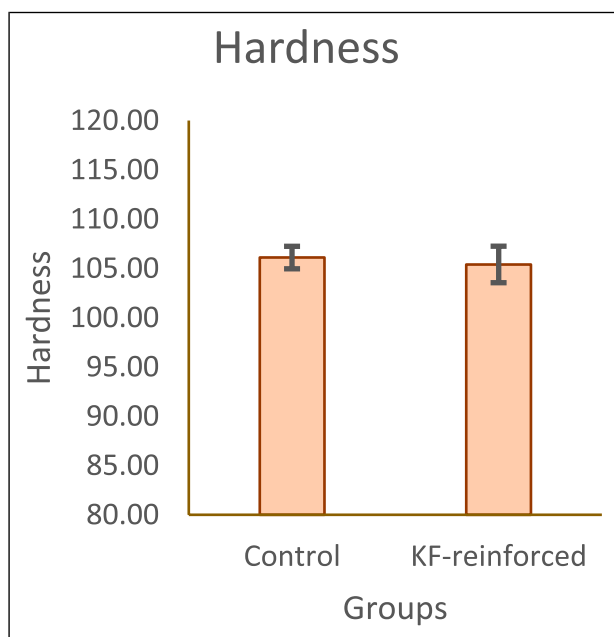


Figure 9. Surface hardness mean value.

strength showed a significant reduction. In the earlier study, both compressive and flexural strengths decreased upon the addition of 1 wt% of alkaline-treated fibers. This contrast suggests that the alkaline treatment may have adversely affected the intrinsic strength of the fibers or altered their surface characteristics, thereby weakening the interfacial adhesion with the nylon matrix. The untreated fibers may have retained better structural integrity, contributing to the observed improvement in compressive behavior. This study differs from our previous work not only in the use of untreated fibers but also in its focus on evaluating the thermal response and mechanical optimization through the modified reinforcement approach, which were not addressed previously.

According to a study conducted by Girdis and associates, macadamia nutshell fibers-reinforcement on acrylonitrile butadiene styrene (ABS) FDM printing exhibited lower compressive strength compared with neat ABS polymer. They suggested that sample design and infill percentage would affect the compressive strength of the printed component as they could generate large voids and slender internal structures unable to accommodate the applied loads.²⁶ It has been reported in the literature that increased kenaf fibers concentration in PLA filament results in decreased flexural strength. This might be attributed to the fiber-fiber bonding rather than fiber-matrix bonding resulting in weak stress distribution in the PLA composite. It was also hypothesized that higher kenaf fibers concentration leads to more fibers agglomeration as a result of physicochemical incompatibility between the hydrophilic cellulosic fibers and the hydrophobic nature of polymers. This agglomeration affects the composite bulk structure by increasing its

porosity which weakens its mechanical strength.²⁷ To mitigate fiber agglomeration, methods such as improving fiber dispersion through optimized mixing techniques, using surface treatments or coupling agents to enhance fiber-matrix adhesion, and controlling fiber length and concentration are effective. According to a study conducted by Arumaiselvan and associates, it was found that natural fibers reinforcement for PLA improves the composite surface hardness significantly compared with neat PLA.²⁸

Based on the study's findings, untreated kenaf fibers can slightly improve compressive strength without changing surface hardness. However, they negatively impact the flexural strength of FDM 3D-printed parts. Thus, the choice to use this reinforcement method largely depends on the specific application and the type of load the printed component needs to endure. Regarding dental prosthesis, compressive strength is crucial for short span denture because it must withstand biting forces concentrated in a small area without fracturing. However, bilateral denture designs face different stresses due to their length and how forces are distributed across the dentures. Therefore, flexural strength is more important as it helps distribute stress evenly to prevent failure or fracture. KF-reinforced nylon fabricated via FDM printing appears suitable for unilateral denture designs where the maximum load is applied vertically to the denture base without necessitating load transfer across the dental arch. Increasing fiber content often raises the likelihood of fiber agglomeration, creating more microstructural defects that act as stress concentrators under load, further reducing mechanical performance. Infill percentage, printing speed, and nozzle diameter can significantly influence the outcome of FDM printing quality and thus impact the mechanical performance of the printed composites. In this study, standard parameters with 100% infill were employed to promote optimum fiber-matrix stress distribution and mechanical integrity. Larger nozzle diameters may help optimize printing strength by improving filament extrusion and layer bonding, although this can compromise surface finish and produce a rougher texture.

Conclusion

The main objective of this study was to evaluate the feasibility of combining untreated kenaf fibers with nylon as reinforcing agent for the purpose of FDM 3D printing. Kenaf fiber reinforcement revealed enhanced thermal stability compared with unreinforced nylon. Marginal improvement of compressive strength was observed for the fiber-reinforced 3D-printed composite (49.34 ± 4.02 MPa) compared to the Control (45.02 ± 5.13 MPa). However, reduced flexural strength of the modified composite was reported (23.23 ± 12.65 MPa) relative to the Control (43.14 ± 3.02 MPa). Surface hardness appeared to have no significant alteration after the process of reinforcement (105.4 ± 1.85 for the fiber-reinforced nylon, and $106.1 \pm$

1.14 for the control nylon). These findings suggest that untreated kenaf fiber-reinforced composite at 1 wt% could provide acceptable mechanical behavior in applications require compression and scratching resistance. However, it may not tolerate bending stresses as expected. It is recommended to observe different fiber loading concentrations for future observations.

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Authors contributions

Al-Kaabi A.: Study design, data collection, statistical analysis, and manuscript writing. Abdullah J. Y.: Study design, data analysis, supervision, and reviewing. Johari Y.: Data analysis and visualization. Abdullah A. M.: Data analysis and visualization. Yhaya M. F.: Data analysis and reviewing. Hasbullah N.: Data analysis and reviewing.

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Declaration of conflicting interests

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Data Availability Statement

The data supporting the findings of this study is available from the corresponding author upon reasonable request.

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