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# Enhancing surface properties of FFF 3D printed nylon reinforced with kenaf fibres for dental prosthesis construction: An ANOVA-based analysis

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## Abstract

Fused filament fabrication (FFF) is a printing technology that relies on remelting and modelling physical objects from thermoplastic filament materials. Natural fibres have been studied and used for polymeric reinforcement. Yet, there is limited information in the literature about reinforcing filament materials for FFF printing. This study aimed to evaluate some surface properties of FFF 3D printed polyamide 6 reinforced with kenaf fibres. Kenaf fibres were retted, bleached with sodium hypochlorite solution, and silanised with 3-aminopropyltriethoxysilane solution prior to thermally compounding with polyamide beads to produce filaments for experimental groups. A total of 55 specimens were printed by an FFF printer for 5 groups: control, 0.1%, 0.3%, 0.5% and 1% kenaf fibres-reinforced polyamide (11 specimens for each group). Fourier-transform infrared spectroscopy analysis was conducted on the control and 1% kenaf-reinforced sample for chemical characterisation. The study groups were submitted to surface roughness, surface hardness and water contact angle measurement tests. The results showed no significant chemical change in the composite as a result of fibre incorporation. Surface hardness have shown a significant increase in their mean values after fibres incorporation, which were 93.3, 93.5, 92.2, 91.3 for 0.1, 0.3, 0.5 and 1% respectively compared with the control 88.9, while there was no significant difference in both surface roughness and surface hydrophilicity except for 1% kenaf-reinforced group (roughness = 358, contact angle = 55.1) compared to the control group (roughness = 216, contact angle = 46.4). Kenaf fibres reinforcement with polyamide 6 at concentrations not more than 0.5% has improved the surface hardness of the FFF printed material.

## Keywords

FFF, polyamide 6, kenaf fibres, reinforced polymer, natural fibres

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## Introduction

Three-dimensional printing technology has experienced significant advances that have allowed its application to be involved in a variety of aspects, such as medical, dental and industrial sectors.[1–4] This additive manufacturing, which is also known as a rapid prototyping method, relies on layer-by-layer fabrication to develop physical objects from digitally designed shapes.[5] The significant advancement in digital dentistry has developed new treatment modalities, especially for dental prosthesis construction.[6,7] Several 3D printing methods have been utilised in this field, such as stereo-lithography, digital light projection (DLP), selective laser sintering, polyjet printing and fused filament fabrication (FFF).[8] Despite the popularity of DLP printing for dental prosthesis construction, it possesses a major disadvantage of possible resin shrinkage.[9] FFF printing, on the other hand, relies on polymer extrusion to fabricate the models. This concept of printing is safe as it does not involve a significant chemical reaction, and can be handled by general practitioner, besides being cost effective.[10] FFF printers use filaments made from thermoplastic polymers as feed stock such as polylactic acid (PLA), polyethylene terephthalate glycol, acrylonitrile butadiene styrene, polycarbonate, nylon

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(polyamide), and some other polymers. However, nylon is one of the suitable polymers for the construction of dental prostheses, such as removable partial denture frameworks and temporary restorations, as it possesses suitable mechanical and physical properties.[11,12] A major downside for FFF printing is the possible physical and mechanical weakness, which is mainly attributed to polymer type, printing parameters, intra-layer bonding, inter-layer bonding, and neck growth between filament layers.[13,14] To resolve this issue, carbon fibres and nanotubes, glass fibres, and natural fibres have been incorporated into the polymers as a reinforcing agent.[15–17] Fibres loading at low ratios optimise mechanical strength and elastic modulus, while higher loading ratios increase the composite viscosity, which eventually leads to poor printing quality and sometimes clogs the printing nozzle.[18] Therefore, it is suggested to keep the fibre fraction ratio low in the composite filament for enhanced printing quality.[19]

Kenaf (*Hibiscus cannabinus*) is a tropical plant that can grow up to 5 m. It is considered to be a major source for natural fibres, especially the stem part, which can be extracted and processed for the usage for a variety of purposes.[20] Kenaf stem consists of two types of fibres, the outer bast fibres and the inner core fibres. The bast fibres constitute 25–40% of the total fibre content, while the inner core fibres represent 60–75% of the stem's total fibre content.[21,22] The Kenaf plant is mostly cultivated in China, India, and several parts of Southeast Asia.[23] Fillers and fibres have been used for polymer reinforcement to promote its mechanical and physical features.[24–26] Recently, natural fibres, such as kenaf fibres, have taken precedence over synthetic fibres for polymer reinforcement for several reasons, including availability, lower cost, and their lower impact on the environment since they are biodegradable.[27–29] Raw fibres require some chemical treatment to eliminate hemicellulose, lignin, and pectin, leaving only pure cellulosic fibres to be incorporated with the polymer. Usually, this treatment involves the retting process, bleaching, and silanisation processes. Due to the presence of a hydroxyl group, cellulosic fibres are considered highly hydrophilic. Therefore, the silanisation process modifies the fibres to blend with the more hydrophobic polymer matrix.[30]

Kenaf fibres have been used as a reinforcing agent on several polymers such as polypropylene, polystyrene, polyurethane, polyvinyl chloride, high-density polyethylene, polyester, PLA, epoxy and composite materials. It was found that kenaf core fibres act as a shock absorber inside the polymer matrix to withstand the applied load, as well as the ability to divert the cracking propagation of the composite matrix, limiting the fracture potential.[31–33] It has also been reported that kenaf core fibres incorporation would improve some physical and surface properties of the composite in terms of tribological properties, water sorption, solubility and moisture content.[34,35]

FFF 3D printing process is based on feeding thermoplastic filament to the printer while it is being heated, melted and deposited through a nozzle on a movable compartment at XYZ directions to build up the physical form layer by layer upon cooling. There is limited information in the literature that indicates the usage of natural fibres, specifically kenaf fibres, as a reinforcing agent for FFF 3D printing. Therefore, this study focused on the assessment of surface enhancement of FFF 3D printed polyamide 6 reinforced with processed kenaf fibres. The assessment was based on chemical characterisation, surface roughness, surface hardness and surface hydrophilicity, which can be achieved through water contact angle measurement.

## Materials and methods

### Materials

In this study, polyamide 6 filament was used for control specimens printing, and polyamide 6 pellets were used for experimental specimens preparation and printing. Kenaf fibres were utilised to reinforce polyamide 6. Sodium hypochlorite was used for fibre bleaching, and 3-aminopropyltriethoxysilane APTES was employed to silanise the fibres.

### Kenaf fibres treatment

Raw kenaf fibres (SKM2-Bio Grade A from National Kenaf and Tobacco Board, Kota Bharu, Kelantan, Malaysia) were water retted for 24 h and then washed and air dried at room temperature for 48 h. Then, the fibres were submerged in 6% sodium hypochlorite solution (FAS, Iraq) for bleaching and further elimination of lignin, pectin, and hemicellulose, and then washed with deionised water thoroughly and left to dry in an oven at 40 °C for 24 h. The last step was the silanisation process, which was conducted by adding the dried fibres to 1:4 ratio solution of ethanol and deionised water containing 0.2 vol.% of APTES (Bide Pharmatech Ltd, China) and stirred for 24 h at 40 °C. After that, the fibres were washed with deionised water carefully and dried in the oven at 40 °C for 24 h. Figure 1 illustrates the fibres before and after sodium hypochlorite treatment.

### Filament production and specimens printing

The fibres were ground and filtered into finer particles with an average diameter of about 477 μm. The fibres were added to polyamide pellets (QIPLAS, China) at the predetermined weight ratios based on the study groups. The mixtures were then applied to the filament extrusion machine (Figures 2 and 3) at a temperature of 250 °C to produce the fibres-reinforced filaments as illustrated in Table 1. Polyamide 6 filament (Torewell™, China) was used for printing the control group's specimens. Ender 3 Neo 3D printer Creality, China, was used for specimens printing, the specimens' dimensions were bar-shaped 100 mm length, 10 mm width, and 3 mm thickness, and designed by Designspark Mechanical 6.0.3 software as shown in Figure 4. The Surface Tessellation Language (STL) file was sliced by PrusaSlicer 2.6.1 software before being transferred to the printer. The printing specifications were illustrated in Table 2.

### Material characterisation and testing

Kenaf fibres were analysed by Fourier-transform infrared spectroscopy (FTIR) to analyse the chemical characteristics of the fibres before and after the bleaching process.

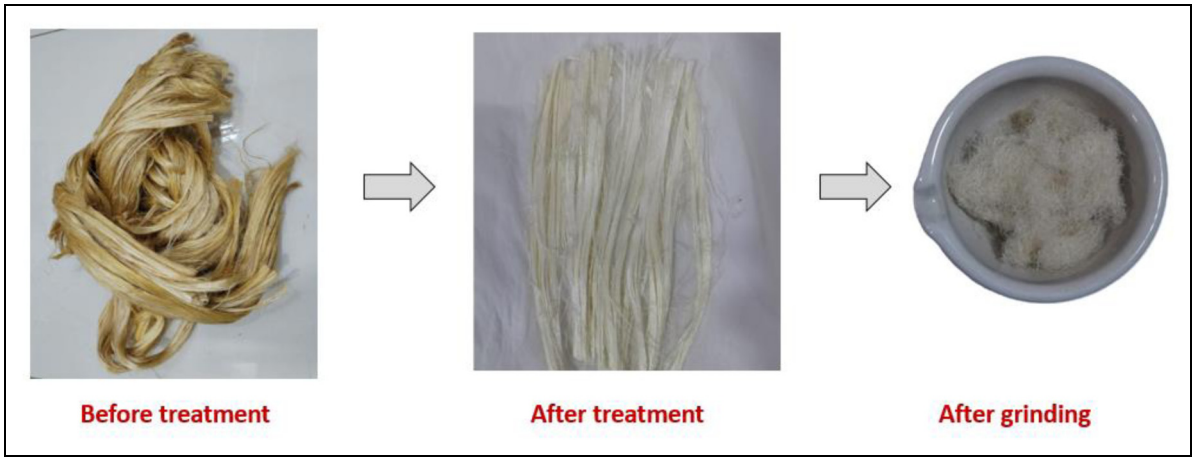


**Figure 1.** Kenaf fibres treatment and preparation.

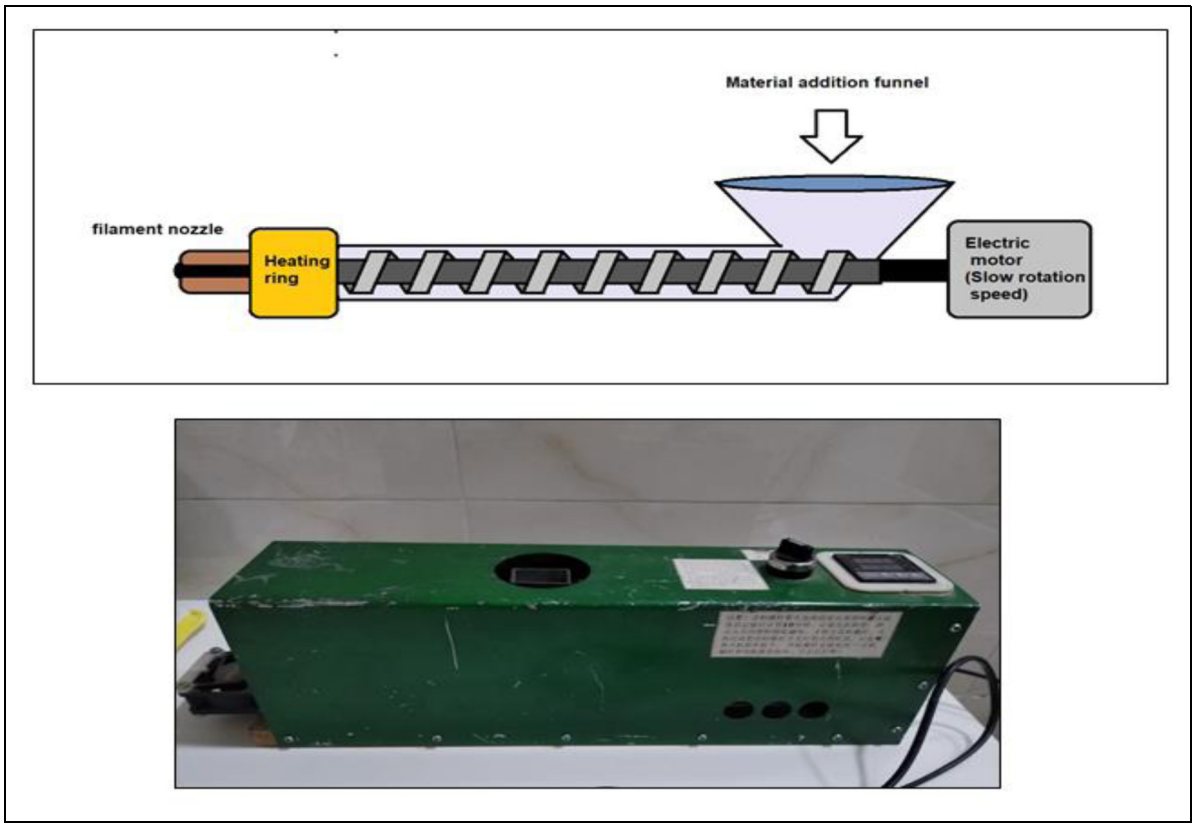


**Figure 2.** Thermal extruding machine.

Fibres-reinforced composite was also analysed for significant chemical differences. The transmission spectra were analysed carefully in terms of band number differences and what they represent in a qualitative manner. Surface properties were observed for the study groups in terms of surface roughness by using a portable profilometer (SRT-6200S, China) by calculating roughness average (Ra). Five different measurements were recorded for each specimen, and the average value was calculated for the statistical analysis. Surface hardness was conducted by Shore A hardness tester (Shantou Yq Technology Co. Ltd, China). Three different measurements were recorded, and the average value was calculated as well. Water contact measurement was conducted by placing a droplet of distilled water with a diameter less than 2 mm on the specimen surface. A digital microscope (Digimicro, China) was used to capture an image of the droplet, which was then processed by ImageJ 1.53e, National Institutes of Health software to calculate the contact angle. Two angles were calculated from both sides of the water droplet, and an average value was calculated for the statistical analysis to avoid any receding or advancing effect of the droplet during placement on the surface, as shown in Figure 5.

## Results

### *FTIR analysis*

As shown in Figure 6, it can be clearly noticed the cellulosic compound by the prominent bands that were similar between

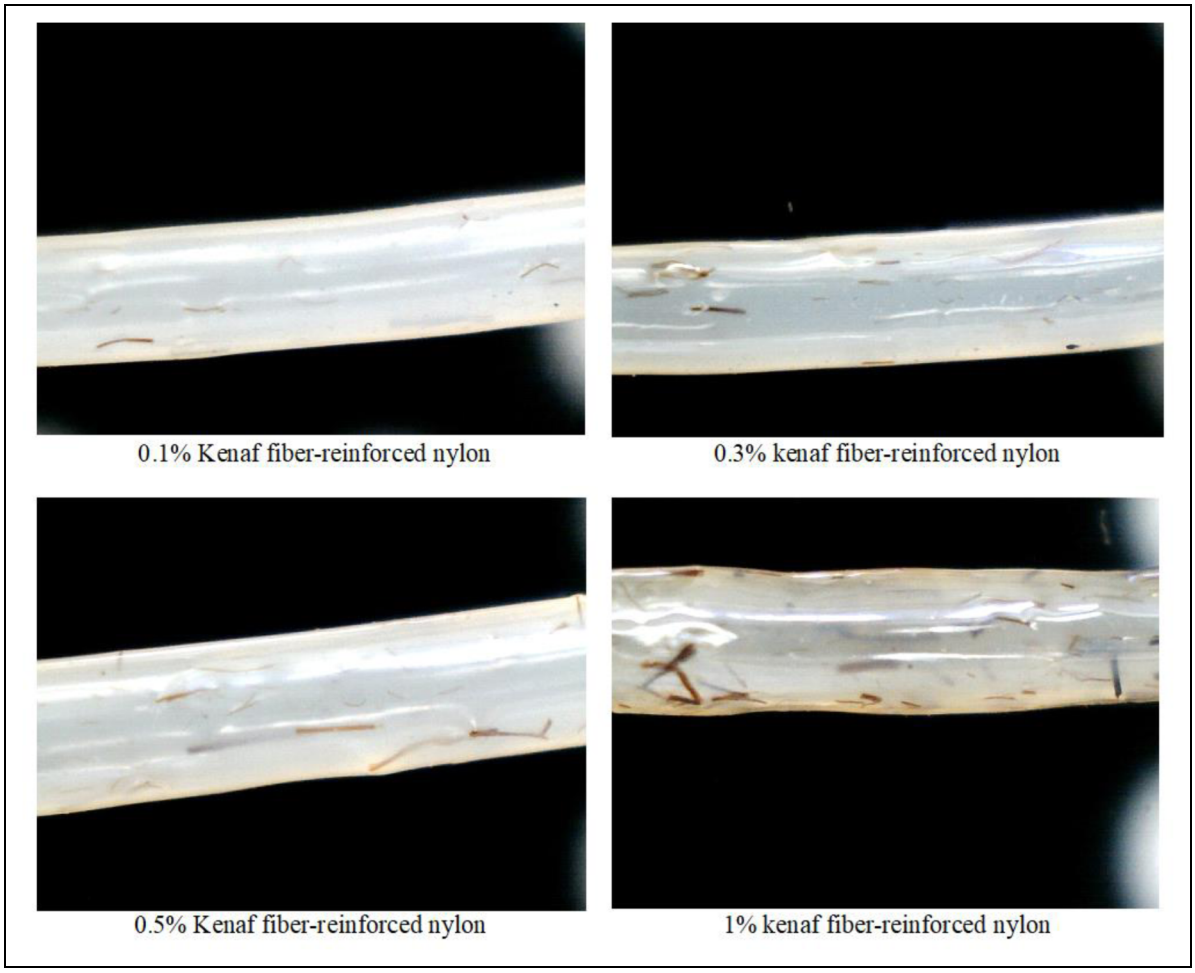


**Figure 3.** Kenaf fibres-reinforced polyamide filaments for the study groups (0.1%, 0.3%, 0.5%, and 1% kenaf fibres-reinforced polyamide filaments).

the untreated and bleached kenaf samples which were 2900 $cm^{-1}$ that represents C-H bond for methyl groups, 1730 $cm^{-1}$ which represents C = O, 1630 $cm^{-1}$ that represent C = C, and several bands from 1300–1460 $cm^{-1}$ that confirms the C-H bonds. There was a minimally distinct band in the untreated kenaf at wavelength 1500 $cm^{-1}$, which represents aromatic rings that disappeared after the bleaching process. Comparing the control sample with the kenaf fibre-reinforced sample, the main polyamide bands are vividly observable, particularly amide I (C = O band at 1640 $cm^{-1}$) and amide II (N-H band at 1540 $cm^{-1}$). This was associated with more prominence in the band at wavelength 1700 $cm^{-1}$ which is relevant to the cellulosic content of the modified polyamide, as shown in Figure 7.

**Table 1.** Fibre preparation and filament extrusion conditions.

| Parameter | Description/value |
|---|---|
| Fibre preprocessing | Fibres were ground and filtered into finer particles. |
| Average fibre diameter | ≈ 477 µm |
| Polymer matrix | Polyamide pellets (QIPLAS, China) |
| Fibre addition method | Thermal mixing |
| Extrusion equipment | Filament thermal extrusion machine |
| Extrusion temperature | 250 °C |

## *Surface analysis*

The descriptive statistics for surface analysis tests are shown in Table 3 and Figure 8. One-way ANOVA test results, as illustrated in Tables 4 and 5, for surface roughness showed no significant difference between control and 0.1%, 0.3%, 0.5% kenaf-reinforced groups (*P*-value >0.05), while there was a significant difference between the control and 1% kenaf-reinforced group (*P*-value <0.05). There was a significant increase in mean hardness values for the kenaf-reinforced groups compared to the control group

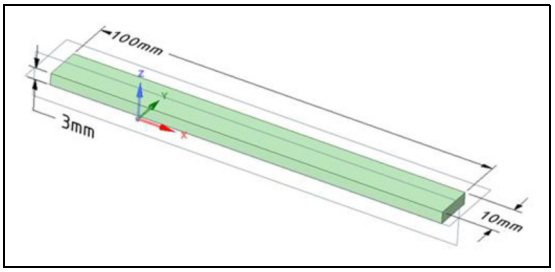


**Figure 4.** Specimen's dimensions for the study groups for FFF 3D printing.

**Table 2.** 3D printing specifications.

| Printing specification | Value |
|---|---|
| Printing speed | 50 mm/s |
| Infill percentage | 100% |
| Nozzle diameter | 0.4 mm |
| Nozzle temperature | 250 °C |
| Bed temperature | 90 °C |
| Layer thickness | 0.2 mm |

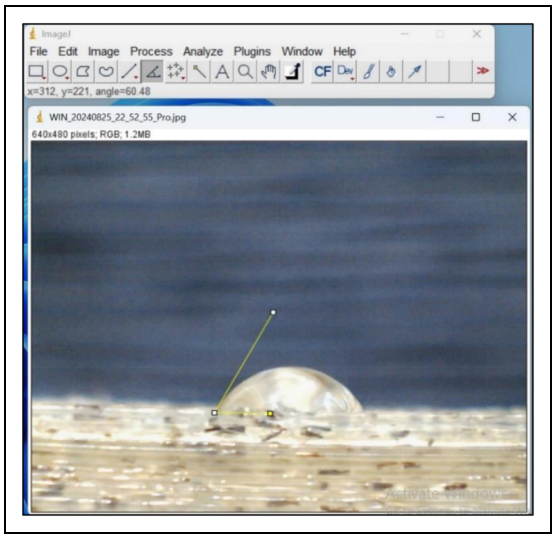


**Figure 5.** Contact angle measurement by ImageJ software.

($P$-value <0.05). The highest mean value was observed for 0.3% kenaf-reinforced group followed by 0.1%, 0.5%, and 1% kenaf-reinforced groups, respectively. Surface hydrophilicity or water contact angle results showed no significant difference in their mean values between control groups and 0.1%, 0.3%, 0.5% kenaf-reinforced groups ($P$-value >0.05), while there was a significant increase in the water contact angle mean value for the 1% kenaf-reinforced group ($P$-value <0.05).

## Discussion

To achieve proper bonding between kenaf fibres and the polymer, the fibres were cleaned, bleached, and then silanised to condition the cellulosic fibre surface and promote its thermal resistance, as it required to be processed at an elevated temperature of 250 °C during filament production and printing. After filament production for the experimental specimens, the fibres appeared to be unidirectionally oriented as previously shown in Figure 3. It is suggested that using a single-screw or twin-screw extruder for filament production may not be sufficient for homogenously mixing the fibres in the polymer matrix. This explains the fibres' disorientation that can be noticed in the higher concentration group.[36]

Chemical analysis by FTIR spectroscopy for kenaf fibres showed no significant change in the chemical composition as a result of the bleaching treatment. The only noticeable difference was the disappearance of the band No. 1500 $cm^{-1}$, which indicates the aromatic rings related to lignin compounds. Moreover, polyamide IR analysis with and without kenaf fibre reinforcement showed no significant chemical change except for the higher prominence of the carbonyl band at a wavelength of 1700 $cm^{-1}$ that is related to the cellulosic content of the reinforced nylon sample. Based on these findings, kenaf fibres appeared to be chemically compatible with polyamide filament material in terms of chemistry. The findings also suggest that the following treatment protocol would not affect the quality of both kenaf fibres and the reinforced polyamide in terms of chemical constitution. These results align with a study conducted by Almeida and his research team, who concluded that natural fibres chemical treatment has modified the fibres properties to be incorporated with PLA filament by reducing the low molecular weight organic components such as lignin and hemicellulose.[37]

Surface evaluation for this study was based on three parameters, which were surface roughness, surface hardness, and water contact angle measurement. Surface roughness evaluation indicated that kenaf fibres reinforcement of polyamide at concentrations equal to or below 0.5 wt% would not affect the surface texture of the printed material at a significant level. Therefore, higher concentrations would result in more surface irregularities as was recorded for the 1% fibres-reinforced group. According to Bellehumeur et al.,[38] natural fibres incorporated during filament production at relatively higher concentrations would result in increased surface irregularities. Due to the buildup of thermal and mechanical stresses as well as heat and mass transfer during the filament extrusion process, fibres aggolmeration and bonding between individual fibres can possibly result.

Surface hardness is a measure of a material's resistance to scratches and indentation. Polyamide falls in the flexible polymers category.[39] For this reason, Shore A surface hardness test was conducted in this study. The results showed a significant increase in surface hardness for the polyamide after kenaf fibre reinforcement for all studied weight concentration groups. These results suggest that fibre incorporation has modified the elastic property and flexibility of the polymer. The surface hardness results agree with a study conducted by Adediran and his associates[40] whose findings showed that kenaf fibres reinforcement would significantly increase polypropylene surface hardness.

Water contact angle reflects the relation between the solid material surface and water. In other words, it is the solid material surface's ability to spread the water droplet on its surface. Surface hydrophilicity is an important property for biomaterials, especially when they are used to construct intra-oral appliances, since a large percentage of the saliva is water. The water contact angle measurements showed no significant change after kenaf fibres incorporation for all studied weight concentrations except for the 1% kenaf fibres-reinforced group. Surprisingly, the 1% fibres-reinforced group showed higher mean contact angle measurements, which indicates less surface hydrophilicity than the remaining studied groups. This finding was unexpected due to the hydrophilic nature of the cellulosic fibres and the hydrophobic nature of the polymer in general.[41] However, according to Haryati and his associates,[30] alkaline treatments and the silanisation process would minimise the hydrophilic nature of kenaf cellulosic fibres.

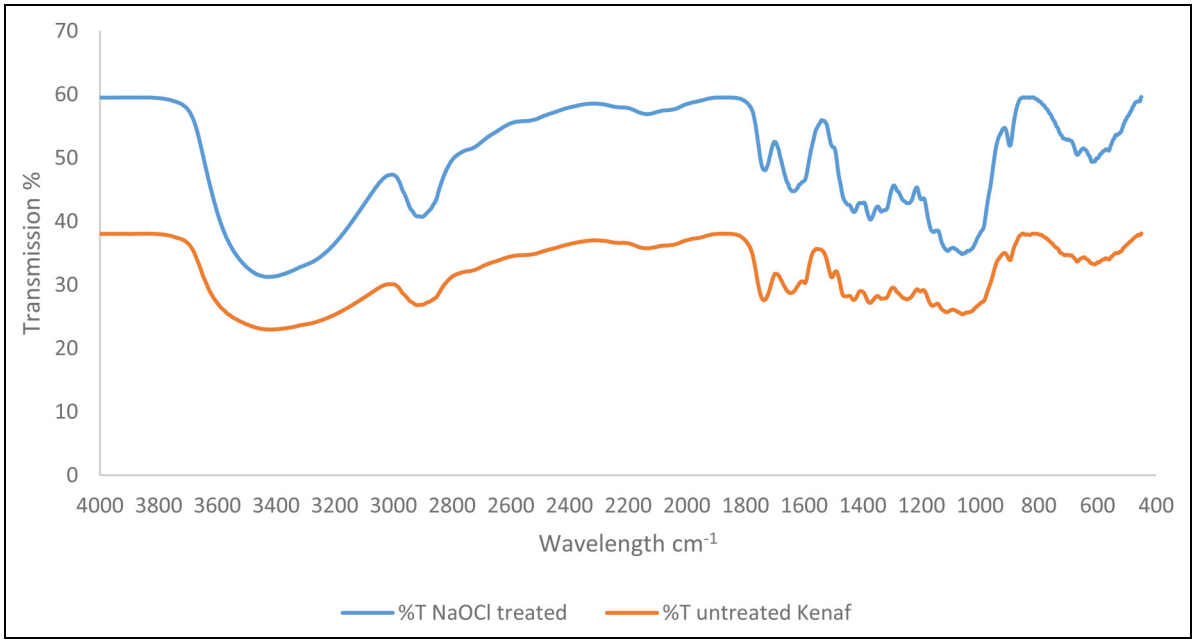


**Figure 6.** FTIR analysis for kenaf fibres before and after treatment with sodium hypochlorite.

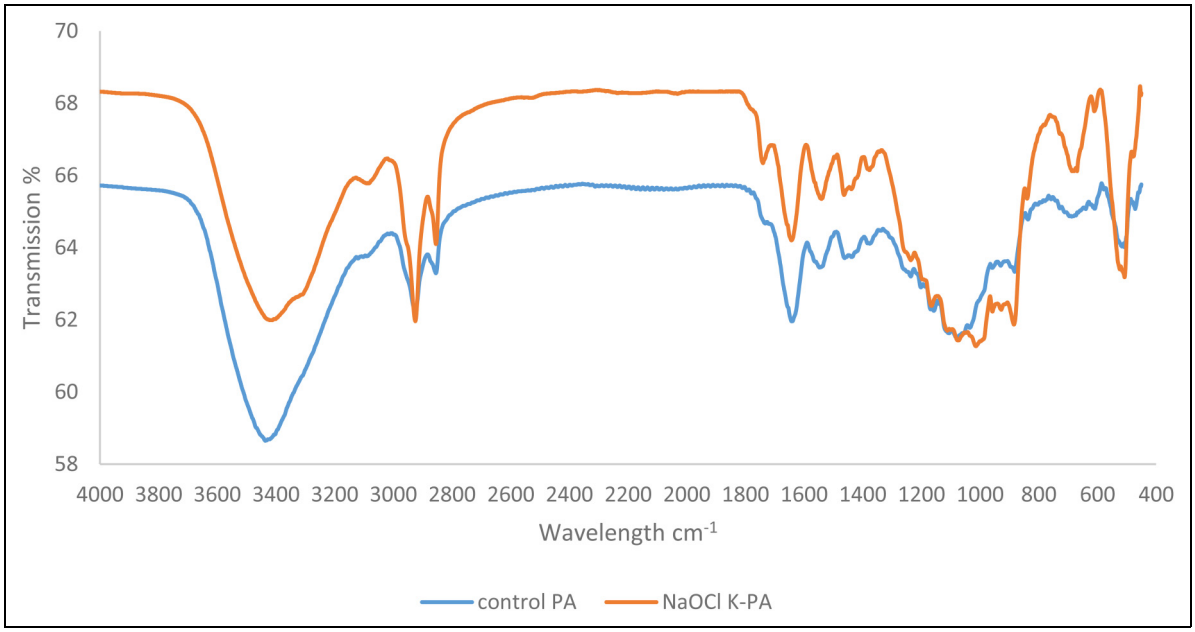


**Figure 7.** FTIR analysis for polyamide before and after reinforcement with treated kenaf fibres.

**Table 3.** Descriptive statistics for surface roughness, surface hardness and water contact angles data.

| | | Surface roughness | | Shore A hardness | | Contact angle | |
|---|---|---|---|---|---|---|---|
| Groups | N | Mean | Std. deviation | Mean | Std. deviation | Mean | Std. deviation |
| Control | 11 | 216 | 97 | 88.9 | 1.7 | 46.4 | 4.6 |
| 0.1% kenaf-reinforced | 11 | 193 | 81 | 93.3 | 1.3 | 44.1 | 3.7 |
| 0.3% kenaf-reinforced | 11 | 151 | 104 | 93.5 | 2.1 | 50.1 | 8.1 |
| 0.5% kenaf-reinforced | 11 | 197 | 87 | 92.2 | 1.1 | 50.3 | 4.7 |
| 1% kenaf-reinforced | 11 | 358 | 66 | 91.3 | 1 | 55.1 | 7.1 |
| Total | 55 | 223 | .11 | 91.8 | 2.2 | 49.2 | 6.8 |

Based on the previously discussed findings, kenaf fibre reinforcement to polyamide 6 filament material at concentrations not exceeding 0.5 wt% would optimise surface hardness of the FFF 3D printed models without affecting their surface roughness and water contact angle. It is highly recommended to further study the composite

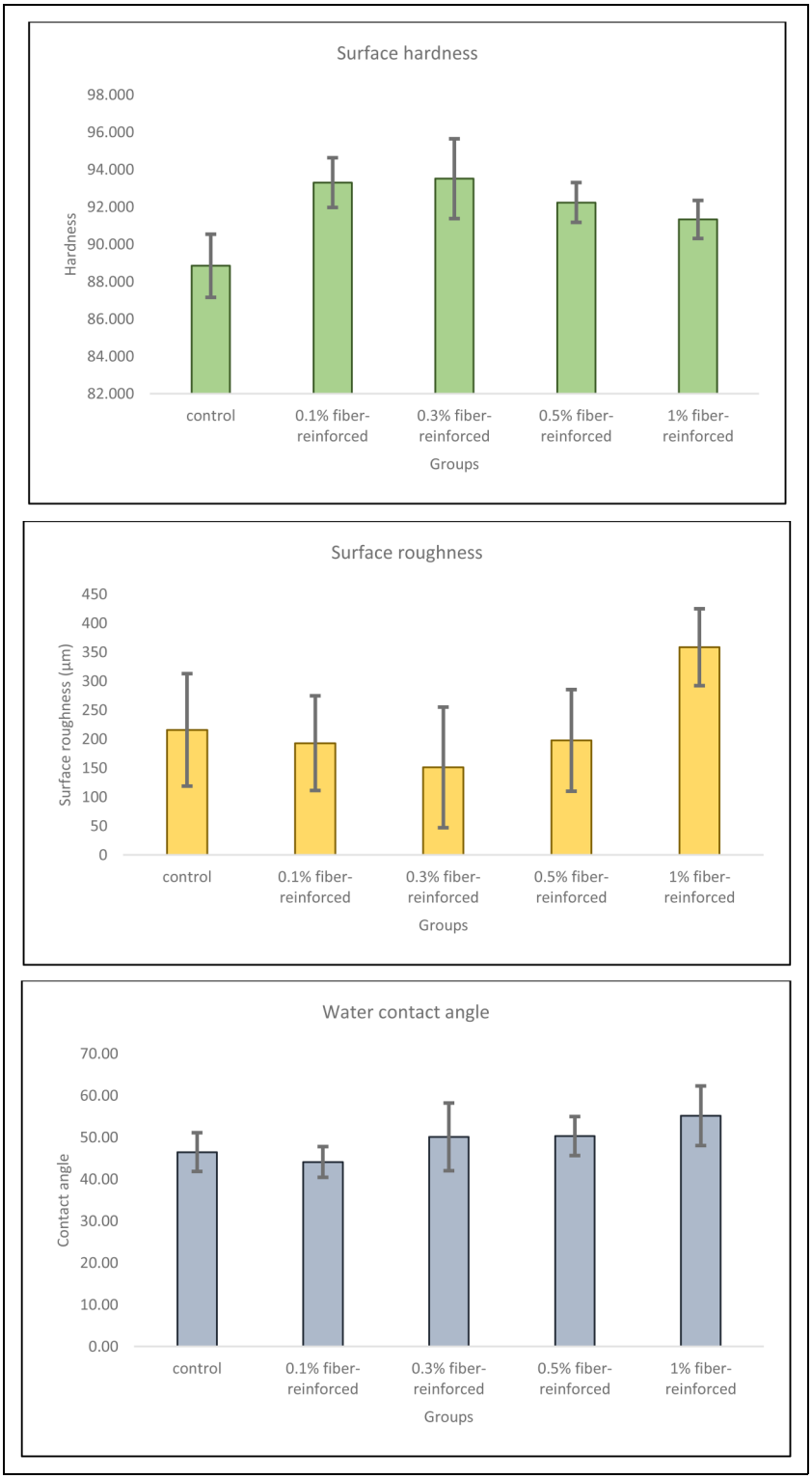


**Figure 8.** Descriptive chart for surface tests mean values.

strength in terms of fracture resistance and strain for future studies.

## Conclusion

Within the limitations of this study, the incorporation of kenaf fibres into polyamide 6 filaments for FFF 3D printing demonstrated a clear benefit at low weight fractions. Reinforcement at or below 0.5 wt% enhanced surface hardness without introducing significant changes in surface roughness or water contact angle, suggesting an optimal balance for dental prosthesis applications where durability, smoothness and wettability are critical for clinical success. FTIR analysis confirmed the chemical compatibility of treated kenaf fibres with polyamide 6, indicating that the pretreatment protocol (retting, bleaching, silanisation) did not adversely affect fibre–matrix interaction. The findings from this study indicate that kenaf-reinforced polyamide 6 filaments at controlled low concentrations offer a promising and sustainable alternative for additive manufacturing of

**Table 4.** ANOVA test results for surface tests.

| | | Sum of squares | df | Mean square | F | Sig. |
|---|---|---|---|---|---|---|
| Roughness | Between groups | 276309. 5 | 4 | 69077. 4 | 8.9 | <0.001 |
| | Within groups | 390324. 5 | 50 | 7806.5 | | |
| | Total | 666634 | 54 | | | |
| Hardness | Between groups | 157.4 | 4 | 39.3 | 17.3 | <0.001 |
| | Within groups | 113.6 | 50 | 2.3 | | |
| | Total | 270.9 | 54 | | | |
| Contact angle | Between groups | 779.5 | 4 | 194.9 | 5.6 | 0.001 |
| | Within groups | 1731.5 | 50 | 34.6 | | |
| | Total | 2510.9 | 54 | | | |

**Table 5.** Tukey HSD for multiple comparisons of surface test data.

| | Groups | N | Subset for alpha = 0.05 | | |
|---|---|---|---|---|---|
| Surface roughness | | | 1 | 2 | 3 |
| | 0.3% kenaf-reinforced | 11 | 150.9 | | |
| | 0.1% kenaf-reinforced | 11 | 192.7 | | |
| | 0.5% kenaf-reinforced | 11 | 197.5 | | |
| | Control | 11 | 215.6 | | |
| | 1% kenaf-reinforced | 11 | | 358.3 | |
| | Sig. | | 0.4 | 1 | |
| Surface hardness | Groups | N | Subset for alpha = 0.05 | | |
| | | | 1 | 2 | 3 |
| | Control | 11 | 88.8 | | |
| | 1% kenaf-reinforced | 11 | | 91.3 | |
| | 0.5% kenaf-reinforced | 11 | | 92.2 | 92.2 |
| | 0.1% kenaf-reinforced | 11 | | | 93.3 |
| | 0.3% kenaf-reinforced | 11 | | | 93.5 |
| | Sig. | | 1 | 0.6 | 0.3 |
| Contact angle | Groups | N | Subset for alpha = 0.05 | | |
| | | | 1 | 2 | 3 |
| | 0.1% kenaf-reinforced | 11 | 44.1 | | |
| | Control | 11 | 46.4 | | |
| | 0.3% kenaf-reinforced | 11 | 50.1 | 50.1 | |
| | 0.5% kenaf reinforced | 11 | 50.3 | 50.3 | |
| | 1% kenaf-reinforced | 11 | | 55.1 | |
| | Sig. | | 0.1 | 0.3 | |

dental prostheses. With expanded mechanical, biological and microstructural validation, this material could significantly advance the field of digital dentistry by providing cost-effective, environmentally friendly and clinically viable alternatives to conventional prosthetic materials.

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## Declaration of conflicting interests

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

## Data availability statement

The datasets generated and analyzed during the current study are not publicly available, but they are available from the corresponding author upon reasonable request. Interested researchers may contact the authors to obtain access to the data supporting the findings of this study.

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