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Comparative evaluation of two bleaching solutions on Kenaf fiber-reinforced nylon: Surface properties and FTIR analysis

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Abstract. 3D printing has revolutionized various industries as well as enriching different important sectors including medicine and dentistry. Fused Deposition Modeling (FDM) is one of the significant techniques for 3D printing which utilizes thermoplastic filament feedstock. Natural fibers have been integrated into composite filament to enhance mechanical and physical defects of the FDM printed materials. The aim of this study was to assess the impact of two types of chemical treatments on kenaf fibers reinforcing polyamide composite filament by evaluating their chemical and surface properties. Sodium hydroxide (NaOH) and sodium hypochlorite (NaOCl) were the two chemical treatment solutions where kenaf fibers was processed at 6% concentration of each solution. Then, the fibers were compounded with nylon beads to form FDM filaments. Three groups of specimens were printed by FDM printer which were control, NaOH-treated, and NaOCl-treated. The samples were characterized by FTIR and evaluated for their surface hardness and surface roughness. The FTIR results showed that NaOH treatment was not as effective as NaOCl treatment in terms of eliminating lignin and hemicellulose. Surface hardness and surface roughness demonstrated minor improvement for the NaOCl-treated group compared to NaOH-treated group. It is recommended to rely on NaOCl treatment at 6% concentration with treatment time not exceeding 24 hours.

Keywords: FDM; Kenaf fibers; NaOH; NaOCl; nylon filament; nylon reinforcement;

1. Introduction

Additive manufacturing technology represented by 3D printing techniques are recently becoming prevailed over the traditional approach for dental prosthesis construction since this technology has the potential to rapidly construct fine and complex dental prostheses following layer by layer building strategy which allow dental practitioners to expand their freedom in formulating the required treatments with minimum procedural steps required (Rezaie *et al.* 2023). Some of 3D printing technologies such as FDM, SLA, SLM and inkjet printing have already started to have a

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significant impact in the production of dental prostheses (van Noort 2012, Shahrubudin *et al.* 2019). FDM is one of the simplest techniques for 3D printing because it is based on thermoplastic polymer extrusion. Fused deposition modeling FDM printing process relies on liquefying and solidifying polymeric filament material into predesigned models (Deng *et al.* 2017, Deng *et al.* 2018, Guo *et al.* 2022).

Nylon (polyamide) is one of the popular filament materials used for printing dental models, appliances and prostheses due to several mechanical and physical properties (Spintzyk *et al.* 2021, Zeidan *et al.* 2023, Al-Kaabi 2025). Some literature reported some mechanical and physical defects that are related to FDM printed models, which were mainly about flexural strength and dimensional stability (Park *et al.* 2021, Freitas *et al.* 2023). Fillers as well as natural and synthetic fibers have been used to reinforce polymers and composites (Garigipati and Malkapuram 2020, Alvarez-Carrizal *et al.* 2021, Muhsin *et al.* 2022, Kati 2022, Jiang *et al.* 2024). Natural fibers are preferred over synthetic fibers for polymer reinforcement for several reasons such as availability, low cost which is about 50% less than synthetic fibers, in addition to their lower impact on the environment (Sayeed *et al.* 2023, Sakhare and Borkar 2022, da Silva *et al.* 2022, Silva *et al.* 2021, Joshi 2004).

Kenaf plant is one of the rich sources of natural fibers that are used for polymers and composites reinforcement (Sapiai *et al.* 2020, Prabhudass *et al.* 2022). The Natural fibers main constituent is cellulose. It is responsible for the stiffness and strength of the fibers, so it acts as a framework that preserves the integrity of the fibers form (Mwaikambo and Ansell 2002). Other secondary components of natural fibers which are hemicellulose that has several carbon sugar molecules linked to it, lignin which is an amorphous aromatic compound, and pectin which is a mixture of polysaccharides (Nabi Saheb and Jog 1999). Thermal degradation of natural fibers starts by lignin decomposition at temperature ranges between 60-200 °C, while cellulose decomposes at a temperature of 350 °C (Joseph *et al.* 2003). Therefore, polymer reinforcement with natural fibers requires chemical treatments to eliminate the hemicellulose, pectin, and lignin leaving only pure cellulosic fibers to be embedded in the polymer matrix (Kamarudin *et al.* 2020).

There is limited information available in the literature to support the application of kenaf fibers reinforcement for 3D printing filament materials. Since FDM 3D printing involves thermal process, it is of significant importance to develop an effective natural fibers treatment protocol to avoid thermal degradation. The aim of this study was to evaluate two different chemical treatment protocols for kenaf fibers and their impact on nylon reinforcement for FDM 3D printing technology in terms of chemical properties and surface quality. The treatment types utilized in this study were sodium hydroxide NaOH and sodium hypochlorite NaOCl bleaching techniques.

2. Methods

2.1 Materials

The control specimens were prepared using nylon filament (polyamide) from Torewell™, China. Polyamide pellets (QIPLAS, China) was used for preparing the experimental specimens. Grade A kenaf fibers (SKM2-Bio Grade A) from the National Kenaf and Tobacco Board, Kota Bharu, Kelantan, Malaysia, were selected for this study. Sodium hypochlorite (FAS, Iraq) and sodium hydroxide (Bide Pharmatech Ltd., China) were used for fiber bleaching techniques and 3-aminopropyltriethoxysilane (APTES) from Bide Pharmatech Ltd., China, was used as a coupling agent for the fibers and nylon polymer.

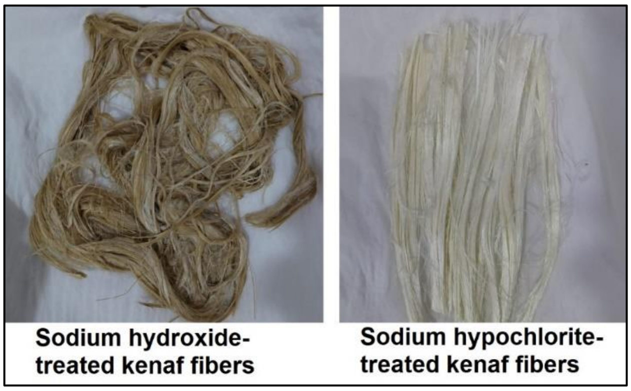

Fig. 1 kenaf fibers after bleaching procedures

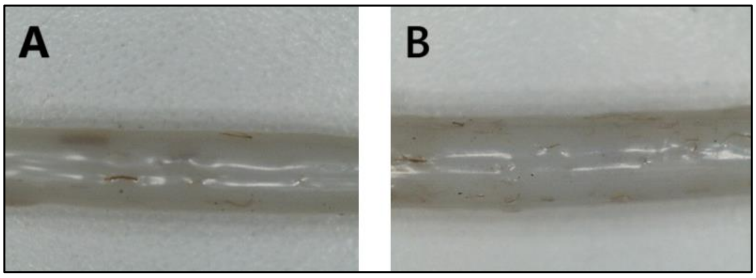

Fig. 2 Kenaf fiber-reinforced nylon filament; (A) sodium hydroxide-treated fibers; (B) sodium hypochlorite-treated fibers

2.2 Kenaf fibers preparation and bleaching

Kenaf fibers were submitted to water submersion for 24 hours and then washed out and left to dry at room temperature for 48 hours. Then, the bleaching procedure was conducted to remove the unnecessary components from the fibers such as lignin, pectin, and hemicellulose. Two samples of fibers were bleached by different solutions. The first sample was added to 6% sodium hydroxide (NaOH) solution for 24 hours, and the other sample was added to 6% sodium hypochlorite (NaOCl) solution for 24 hours. Each sample of fibers was washed with deionized water thoroughly and dried in an oven at 40°C for 24 hours. Fig. 1 shows the fibers form after bleaching process.

Both samples were silanized by 3-Aminopropyltriethoxysilane (APTES) to preserve the fibers during compounding with the polymer at elevated temperature during filament production and to ensure optimum bonding. Each fibers sample was added to 1:4 ratio solution of ethanol and deionized water containing 0.2 vol. % of APTES and stirred for 24 hours at 40°C. The fibers samples were then washed with deionized water and dried for 24 to be ready for the polymer compounding procedure.

2.3 Fiber-modified filament production

The fibers from each sample were ground and Sieved (Retsch sieve shaker) using mesh size 40 (400μm) before adding to nylon pellets at a weight concentration of 0.5%. The composite filaments

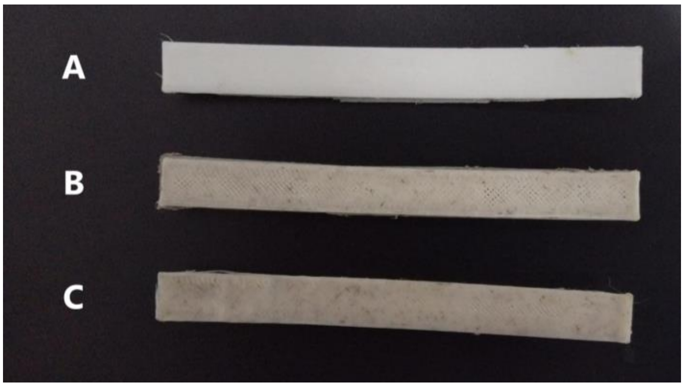

Fig. 3 Examples of the groups specimens: (A) control sample; (B) sodium hydroxide treated sample; (C) sodium hypochlorite treated sample

were produced at a compounding temperature of 250°C. In order to inspect fibers distribution, micro-images were taken for each type of the modified filament and shown in Fig. 2. Specimens were printed by Creality Ender 3 Neo 3D printer, China. The printing speed was set to 100% (50 mm/s), and the in-fill was 100%. The nozzle temperature was 250°C and the bed temperature was 90°C. The specimens' dimensions were 100mm in length, 10mm in width and 3mm in thickness as shown in Fig. 3. The study groups were control, sodium hydroxide-treated, and sodium hypochlorite treated fibers-modified. Each group consisted of 5 specimens.

2.4 Analysis tools

Chemical characterization by Fourier Transform Infrared Spectroscopy FTIR was conducted on the fibers before and after each treatment method. The control and modified filament materials were also characterized using Fourier Transform Infrared (FTIR) spectroscopy. The study specimens were submitted to surface hardness test (Shore A, Shantou Yq Technology Co. Ltd., China), and surface roughness test by portable stylus surface roughness tester (SRT-6200S, China) to inspect the effect of fibers addition on the surface properties of polyamide composite. The statistical analysis was conducted by Using IBM Statistical Package for the Social Sciences SPSS, the data were analyzed for normality and the descriptive statistics were observed for mean values and standard deviations. One-way ANOVA test was conducted for significant difference, and Tukey HSD for multiple comparisons was reviewed for significant difference among the study groups.

3. Results and discussion

Fused Deposition Modeling is a 3D printing technology widely used in medical, dental, and industrial sectors due to its straightforward operation, cost-effectiveness and safety. Thermoplastic polymers are used as filament for FDM printing technology which relies on melting and extruding the polymer into predesigned CAD shapes. Due to some limitations in the physical properties of the FDM 3D-printed materials, synthetic and natural fibers have been added to reinforce the material. Natural fibers required special chemical treatment before being incorporated to the polymer filament. Chemical treatment plays a crucial role in preparing fibers for reinforcement with polymers

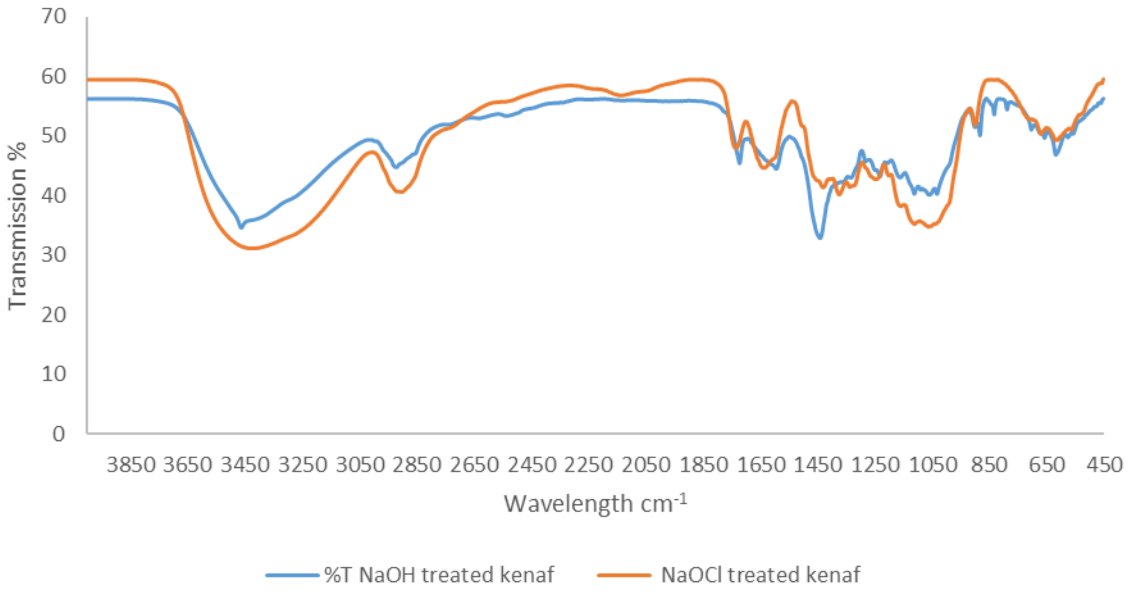

Fig. 4 Kenaf fibers after chemical treatments with NaOH and NaOCl

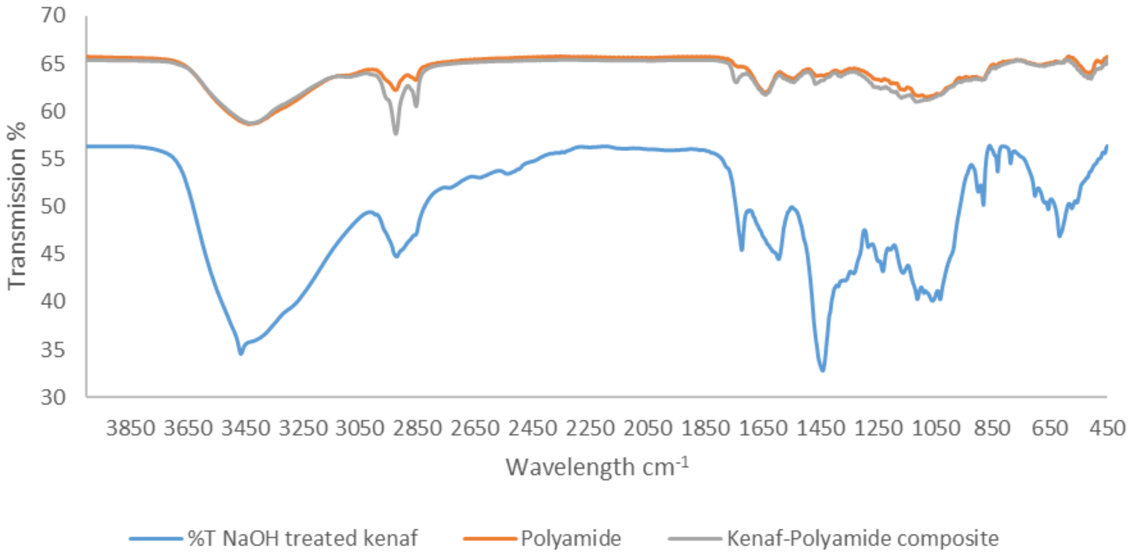

Fig. 5 Sodium hydroxide-treated kenaf –reinforced nylon comparison

and composite. It has been reported in the literature that alkaline treatment for kenaf fibers would possibly cause damaging effect on the fibers and affect their adhesion to polymer matrix (Ismail *et al*. 2021) However, some researcher recommended employing alkaline treatment for short duration and with small concentrations to minimize fibers deterioration while maximizing the benefits of reinforcement to the polymers (Mahjoub *et al*. 2014, Fiore *et al*. 2015). This study aimed to evaluate two distinct treatment methods for kenaf fibers to reinforce Nylon filament, thereby enhancing the physical properties of 3D- printed materials.

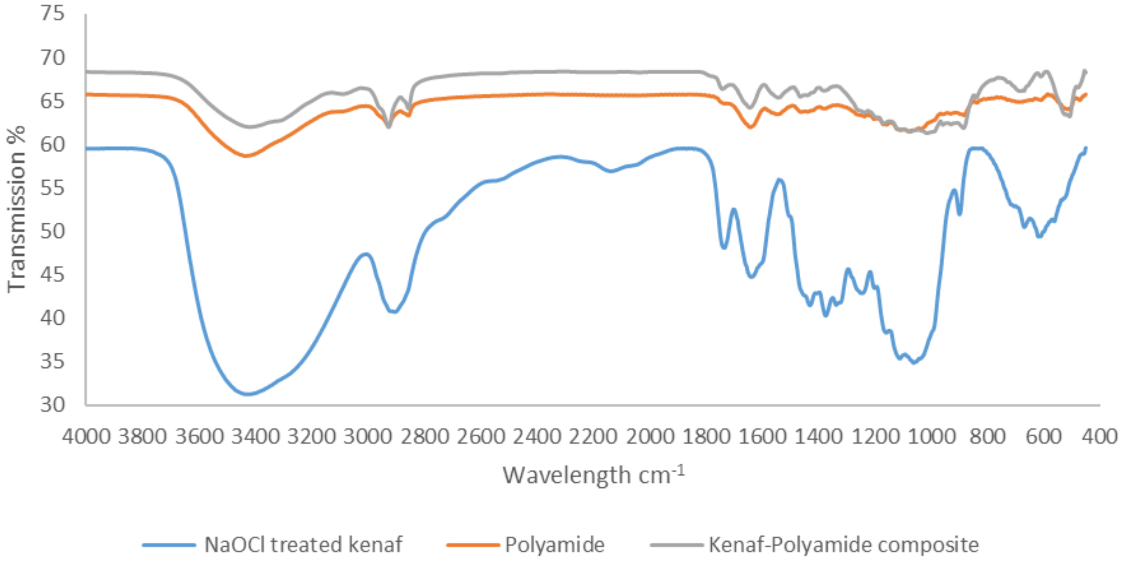

Fig. 6 Sodium hypochlorite-treated kenaf –reinforced nylon comparison

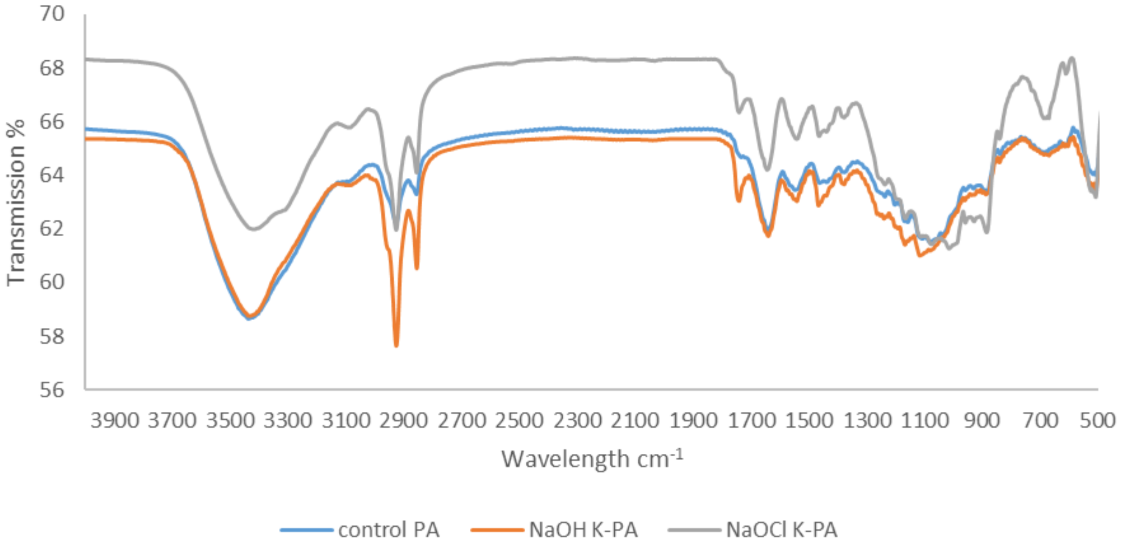

Fig. 7 FTIR analysis for control PA, NaOH-treated K-PA, and NaOCl-treated K-PA

3.1 FTIR spectroscopy analysis

Fourier Transform Infrared Spectroscopy results were analyzed for the significant bands difference for the fibers after treatment with NaOH and NaOCl solutions, for the fiber-modified composites as well. The presence of the functional groups in the data were evaluated and compared. Although these signals assignments might be overlapped, only distinct variations were confirmed (Andrade-Guel *et al*. 2020). Fig. 4 represents the IR spectra for the two fibers treatment samples. The band at wavelength of 1720 cm^{-1} represent the vibration of C=O bond, which refers to ester of

Table 1 Descriptive statistics for shore A hardness results

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
Control	5	108.40	2.074	.927	105.83	110.97	105	110
Sodium Hydroxide-treated	5	107.20	2.280	1.020	104.37	110.03	104	110
Sodium Hypochlorite-treated	5	109.60	1.517	.678	107.72	111.48	108	112
Total	15	108.40	2.098	.542	107.24	109.56	104	112

Table 2 One Way ANOVA tests results for shore A hardness data

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	14.400	2	7.200		
Within Groups	47.200	12	3.933	1.831	0.202
Total	61.600	14			

aldehyde groups. This band seems more prominent in the NaOH treated kenaf fibers sample. Another significantly distinct band is at the wavelength of 1440 cm^{-1} that is found in the NaOH fibers sample spectrum. This band represent the C-H bond vibration. Considering the chemical structures of kenaf fibers components, these NaOH distinct bands are attributed to the presence of lignin along with the cellulose. This finding indicates that NaOH is not as effective as NaOCl solution for fibers bleaching treatment.

Fig. 5 illustrates the spectra for the NaOH treated fibers, nylon, and fiber-reinforced nylon. The composite spectrum clearly confirmed the previously discussed bands as they appear not aligned with the nylon spectrum with more distinction for the bands between 2800 cm^{-1} and 3000 cm^{-1} that stand for the methyl groups bonds vibration. Fig. 6 shows the spectra for the NaOCl treated fibers, nylon, and fibers-reinforced nylon. The band presented by wavelength 880 cm^{-1} usually refers to the C=C bond. However, major bands indicate this chemical bond cannot be observed. Fig. 7 which presents nylon, and the two fibers reinforced nylon composite spectra, a wide band at wavelength ranging between 600 cm^{-1} and 800 cm^{-1} can be observed for the NaOCl fibers treated composite sample. This band ascribes the C-Cl bond. Although bond indicates possible interaction between NaOCl solution and kenaf fibers, their significant effect on the quality of the composite is not yet understood.

3.2 Surface hardness

Surface hardness is a measure of materials resistance to scratches and indentation. It is an important property when it comes to the FDM printing of models and parts as its value affects the durability of the printed material. High surface hardness assures the integrity of the material surface by increasing its resistance to scratches and minimizing wear especially when the printed parts are subjected to mechanical stresses or frequent handling. In this study, Shore A surface hardness test was conducted on the samples and the descriptive statistics as well as the ANOVA test results are reported in Tables 1 and 2.

Table 3 Descriptive statistics for surface roughness

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
Control	5	0.227	0.004	0.002	0.222	0.232	0.223	0.233
Sodium Hydroxide-treated	5	0.250	0.037	0.017	0.204	0.297	0.213	0.292
Sodium Hypochlorite-treated	5	0.189	0.018	0.008	0.167	0.211	0.163	0.212
Total	15	0.222	0.034	0.009	0.203	0.241	0.163	0.292

Table 4 One Way ANOVA tests results for surface roughness

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	8.457	0.005	8.457		
Within Groups	0.007	12	0.001	0.005	8.457
Total	0.017	14			

According to Table 1, surface hardness test results showed slight increase in mean value for the sodium hypochlorite group with the least value for the standard deviation, even though the ANOVA test showed no statistically significant difference among the groups as in Table 2. These results could indicate that sodium hypochlorite fiber-treatment would improve the nylon composite surface hardness compared to sodium hydroxide fiber-treatment. However, it has been reported in the literature that kenaf fibers reinforcement of polylactic acid has significantly increased the composite hardness (Khan *et al*. 2024).

3.3 Surface roughness

Material's surface texture is highly influenced by the form, number, and degree of imperfections, irregularities, and deviations from the perfectly smooth surface. These factors usually refer to the surface roughness. Surface roughness is a critical parameter that affect the functionality, performance, and longevity of components especially when they have been used for biomedical applications such as dental prostheses. Surface roughness assessment was conducted in this study to evaluate the effect of kenaf fibers treatment methods on the final composite surface finish. Table 3 shows the descriptive statistics for the study groups while Tables 4 and 5 show the One way ANOVA test results for significant difference among the study groups. Based on the statistical analysis, there was a significant difference between the NaOCl-treated group and NaOH-treated group ($p\text{-value} < 0.05$) with increased in mean roughness value for NaOH. However, both fiber-modified groups were not significantly different from the control group.

While efficient natural fibers bleaching process eliminates components like lignin and hemicellulose, it is hypothesized that more powerful bleaching compounds would excessively remove these components in addition to changing the form of the cellulose fibers by increasing the pores and etches on their surfaces (Beg *et al*. 2023). These effects can be noticed by the lighter color of the NaOCl-treated fibers compared to the darker colored NaOH-treated fibers. Fibers roughness

Table 5 Tukey HSD test for multiple comparison for surface roughness data

Groups	N	Subset for alpha = 0.05
		1
NaOCl treated group	5	0.18880
Control	5	0.22680
NaOH treated group	5	
Sig.		0.065

improve the bond between the fibers and the resin or polymer as the fibers surface irregularities would provide more grip points for the polymer to enhance the mechanical strength of the composite. However, these fibers irregularities would cause some surface protrusions from the composite that affect its surface texture. Considering the low kenaf fibers concentration (0.5 wt %) used in this study, the fibers texture influence on the composite surface roughness would not be significant.

4. Conclusions

Natural fibers have gained precedence over synthetic fibers for polymer reinforcement due to economic and environmental considerations. The objective of this study was to assess two chemical treatment modalities for kenaf fibers for nylon reinforcement in terms of chemical composition and surface quality. FTIR chemical characterization indicated that sodium hypochlorite would be a better option for kenaf fibers treatment compared to sodium hydroxide. This treatment also resulted in marginal improvement for the composite surface roughness and surface hardness as kenaf fibers reinforcement would produce smoother surface and with increased resistance to handling and scratches compared to the unmodified nylon. Further mechanical and surface analysis for kenaf fibers-reinforced nylon composite are highly recommended for future studies.

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