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Original Research

Chemically Etched 3D-Printed Optical Fibers for Biocompatible Applications

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ABSTRACT

Hydrogel coatings enhance the biocompatibility of optical fibers by reducing mechanical mismatch with tissue, with chemical etching as a critical first step to improve coating adhesion. However, the effects of sodium hydroxide etching on 3D-printed fibers remain understudied. Polyethylene terephthalate glycol (PETG) has shown great promise in advancing 3D printing technology for optical fiber fabrication due to its low optical loss, printability, and chemical resistance. In this study, PETG optical fibers were etched in 0.1 mM and 1 mM NaOH for 30–120 minutes and evaluated for optical loss, morphology, and chemical structure. All fibers maintained low optical loss, with a maximum of 0.18 dB/cm after 2 hours in 1 mM NaOH. No visible degradation or discoloration was observed, and FTIR analysis showed no structural changes at =C–H, CH_2, C–O–C, C=O, and C–H stretching, confirming no degradation at structural level which could compromise the biocompatibility of PETG. These results confirm that mild chemical etching does not compromise the optical or chemical integrity of PETG fibers, supporting their suitability for biocompatible, hydrogel-coated optical systems in biomedical applications.

INTRODUCTION

Optical fibers are extensively utilized in biomedical applications, including bio-sensing, waveguiding, and photoacoustic imaging. Silica-based optical fibers, known for their ultralow transmission loss, remain the industry gold standard. However, their inherent brittleness and limited flexibility pose significant risks of fracture during implantation, rendering them limiting their use in invasive biomedical procedures involving direct tissue contact. Moreover, the ultralow loss characteristic of silica fibers is unnecessary in biomedical contexts, where fiber lengths typically span only a few centimeters. As a result, polymeric optical fibers are increasingly favored for their superior flexibility and intrinsic biocompatibility.(Gierej et al., 2021; Min et al., 2022; Wang et al., 2021).

Despite the advancements of optical fibers in biomedical field, its biocompatible is still hindering its clinical translation. Coating optical fiber with hydrogel has been shown to significantly improve the biocompatibility (Choi et al., 2015; Fujiwara et al., 2020; Zhou et al., 2023). However, coating hydrophilic hydrogel onto the fiber surface require multi-steps procedure to enhance the adhesion between the two surface beginning with chemical etching, followed by silanization and crosslinking (Liu et al., 2021a).

In recent years, 3D printing technology has been widely explored as a new approach for fabricating low cost optical fiber. With the advancement of additive manufacturing, Fused Deposition Modeling (FDM), a widely used 3D printing

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technique has gained increasing attention as a promising alternative for optical fiber fabrication. Unlike the conventional mold casting technique which the process is labor intensive, time consuming, unsuitable for large production and the process itself has been found to increase the attenuation of the optical fiber (Gierej et al., 2023), FDM offers a rapid and continuous production while maintaining a low attenuation using the low cost commercial filaments (Canning et al., 2016; Cook et al., 2016; Hossain et al., 2022; Pires-Junior et al., 2023). When employing commercial filaments for 3D-printed optical fibers, the primary selection criterion is optical transparency, specifically a transmittance exceeding 90%. This requirement significantly limits the range of suitable filament materials.

Among the most commonly used filaments reported in the literature for 3D-printed optical fibers are acrylonitrile butadiene styrene (ABS), polyethylene terephthalate glycol-modified (PETG), and polylactic acid (PLA). In a study by Canning et al. (2016), ABS-based 3D-printed fibers exhibited higher optical attenuation (0.33 dB/cm) compared to PETG fibers (0.26 dB/cm) at a wavelength of 543 nm. A similar trend was observed at 1550 nm, with attenuation values of 0.38 dB/cm for ABS and 0.26 dB/cm for PETG. Beyond passive transmission, PETG has also demonstrated promise as a functional material in sensing applications, with Hossain et al. (2022) reporting successful integration of PETG fibers in a colorimetric sensor (Canning et al., 2016; Hossain et al., 2022). In contrast, a higher attenuation of 0.64 dB/cm at 543 nm was observed in a study by Cook et al. (2016) for a fiber comprising an ABS core and PETG cladding.

Notably, recent advancements have improved the optical performance of 3D-printed PETG fibers, with (Pires-Junior et al., 2023) reporting a remarkably low attenuation of 0.04 dB/cm for a 0.4 mm PETG fiber. In our previous work, we achieved similarly low attenuation values of 0.08 dB/cm for 1 mm-diameter PETG fibers, with the optical loss remaining stable even after prolonged exposure to hydrogen peroxide (Syakirah Mohamad Safri et al., 2024). Collectively, these findings highlight the strong potential of PETG as a cost-effective and optically stable material for 3D-printed optical fiber applications.

Among these steps, chemical etching serves as a critical initial treatment to enhance surface reactivity and subsequent coating adhesion. Commonly employed etching agents include sodium hydroxide, potassium hydroxide, and hydrochloric acid, with fibers immersed for controlled durations (Puygranier & Dawson, 2000; Tao et al., 2010). Sodium hydroxide (NaOH), in particular, has been widely reported in the literature, with concentrations ranging from 0.1 mM to 1.0 mM and immersion durations from 30 minutes to 2 hours (Kang et al., 2023; Multar et al., 2020; Zainuddin et al., 2018). Despite its widespread use, the influence of chemical etching on the optical transmission properties of the fiber remains insufficiently explored, particularly in the context of 3D-printed polymeric fibers. Previous study on chemical resistance of PETG products have reported on degradation of the performance assessed from the impact resistance which dropped significantly to 72% after immersed in 37% hydrochloric acid (Kaptan, 2025). Similar findings have been observed when immersing PETG in acid cider and vinegar where scanning electron microscopy results revealed signs if deformation to the PETG (Carrete et al., 2019). This raises important concern when fabricating optical fiber using the commercial filaments. With the limited studies involved immobilization on sensing region of the fibers, it is essential to optimize both the etching concentration and exposure time to preserve optical performance while enabling effective surface functionalization for providing fundamental insights for future applications. In this study, PETG optical fibers were directly fabricated using FDM 3D printing and subsequently immersed in sodium hydroxide solutions of varying concentrations and durations. The etched fibers were then systematically characterized in terms of optical loss, surface morphology, and chemical structure.

MATERIALS AND METHOD

Material

PolyLite™ polyethylene terephthalate glycol (PETG) 3D printing filament with diameter of 1.75mm in clear color was purchased from Playmaker. Sodium hydroxide from Sigma Aldrich.

Fabrication of 3D printed PETG optical fiber core

The PETG optical fiber core was fabricated following the method established in our previous work. Using a commercially available PETG filament, the core was directly extruded from the nozzle of an Ender 3 S1 Pro 3D printer (Creality Technology, Shenzhen, China). Manual extrusion settings were applied, as illustrated in Figure 1. Initially, the nozzle temperature was set to 200 °C, and a nozzle diameter of 0.8 mm was used (Mohamad Safri et al., 2024; Pires-Junior et al., 2023). To prevent bending or deformation of the extruded fiber core during printing, the distance between the nozzle and the bedplate was maximized. This configuration ensured a straight and uniform extrusion of the fiber core suitable for further processing and characterization.

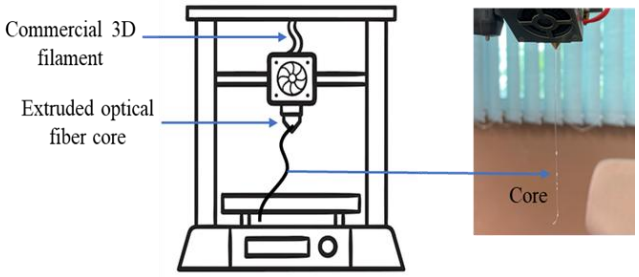

Fig. 1 Fabrication of 3D printed core

Chemical etching in Sodium Hydroxide

Sodium hydroxide (NaOH) solutions were prepared at concentrations of 0.1 mM and 1 mM by diluting NaOH pellets in distilled water under continuous stirring until fully dissolved. The 3D-printed PETG optical fibers (0.8 mm diameter, 10 cm length) were individually immersed in the NaOH solutions contained in chemically resistant borosilicate glass tubes. Etching was performed for 30, 45, 60, and 120 minutes at room temperature (~25°C). All procedures were conducted inside a fume hood within a well-ventilated laboratory, adhering strictly to chemical handling safety protocols to minimize alkaline exposure risks. Following the etching process, each fiber was immediately rinsed under flowing distilled water for 30 seconds

to remove any residual chemical. The fibers were then air-dried for 30 minutes prior to further characterization (Abdul Rashid et al., 2017; Ramakrishnaiah et al., 2016).

Optical loss

Optical loss of the fiber was measured using the cut-back method, where the optical output was recorded using a plastic optical power meter (POFPM100, Shanghai Tarluz Telecom Tech, China) before and after shortening the fiber. The loss was then calculated using the formula in Eq. 1. A 650 nm laser light source (Proskit MT-7615) was connected to one end of the fiber, while the other end was attached to the power meter as shown in Figure 2. Connector losses of 0.3 dB/m were considered negligible due to the short fiber length used (<10 cm). During measurement, the fibers were kept in a straight position to minimize bending loss (Bartolomei, 2021; Fujiwara et al., 2023; Gierej et al., 2019)

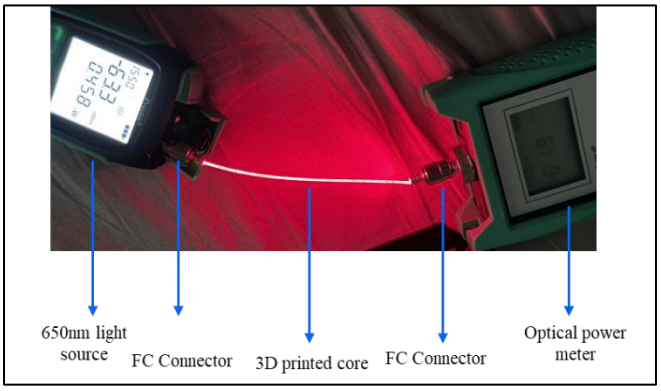

Fig. 2 Setup for the optical loss measurements

$$\alpha = \left| \frac{10}{L_1 - L_2} \log_{10} \frac{P_1}{P_2} \right| \quad [\text{Eq.1}]$$

where,
α- attenuation, in dB/cm, at wavelength
P1- optical power after cut
P2- optical power before cut
L1- Original length
L2- Cut length

Morphology

The surface morphology of the optical fibers was analyzed using an optical microscope at 40× magnification to assess any physical degradation resulting from the chemical etching process. The inspection focused on identifying surface defects such as micro-cracks, pits, or irregularities that could indicate material degradation or structural compromise. Each fiber sample was placed on a clean glass slide and examined along multiple segments of its length to ensure uniformity in surface condition.

Fourier Transform Spectrometer (FTIR)

FTIR spectra of the 3D-printed optical fibers were recorded using a PerkinElmer spectrometer at room temperature, within the wavenumber range of 4000–600 cm^{-1}. The measurements were performed with a resolution of 4 cm^{-1} and averaged over 32 scans to enhance signal-to-noise ratio. Prior to analysis, all fiber samples were carefully cleaned with acetone to remove any surface contaminants or dust that could interfere with the spectral readings. A constant contact force of 40 N was applied across all samples to ensure consistent sample-to-crystal contact during measurement, thereby minimizing spectral variations due to pressure-induced differences in absorbance. This characterization aimed to evaluate any chemical changes in the PETG fiber surface following the etching process (Nassier & Shinen, 2022; Pakarzadeh et al., 2012).

RESULT AND DISCUSSION

Optical loss

Figure 3 presents the optical loss measurements of chemically etched 3D-printed PETG optical fibers. Despite variations in sodium hydroxide concentration (0.1–1.0 mM) and immersion durations (30–120 minutes), all fiber samples exhibited optical losses within the acceptable range of 0.1 dB/cm to 2.6 dB/cm, which is considered suitable for effective light transmission in the visible range of 450–800 nm (Liu *et al*., 2021b). The measured losses are consistent with previous studies on PETG-based optical fibers, such as the work by (Canning et al., 2016), which reported an attenuation of 0.26 dB/cm. These results also align with our earlier findings, where 3D-printed PETG fibers maintained low optical loss even after prolonged exposure to hydrogen peroxide for up to three weeks, further demonstrating the chemical durability and optical stability of PETG under aggressive conditions (Mohamad Safri *et al*., 2024). Maintaining low optical attenuation is particularly critical for applications such as photomedicine, a therapeutic approach that utilizes light (photons) for disease treatment. In these contexts, including fiber-based waveguides, minimizing optical loss ensures sufficient light penetration through biological tissues and accurate delivery to the target treatment site. Therefore, while optimizing the biocompatibility of optical fibers for biomedical applications, preserving low attenuation remains essential to ensure both therapeutic effectiveness and sensor reliability.

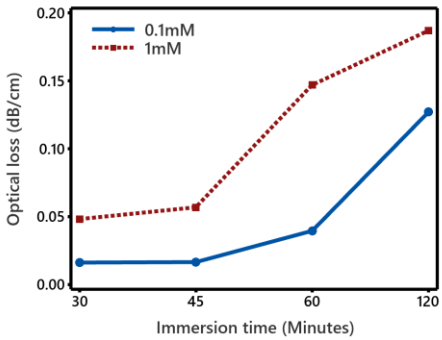

Fig. 3 Optical loss for the etched fibers

Morphology

Results presented in Figure 4 (a) show no visible signs of surface degradation such as cracks, pits, or structural irregularities across all etched fiber samples, including those subjected to the highest sodium hydroxide concentrations and longest immersion durations. Additionally, as illustrated in Figure 4 (b), immersion in sodium hydroxide did not result in any observable

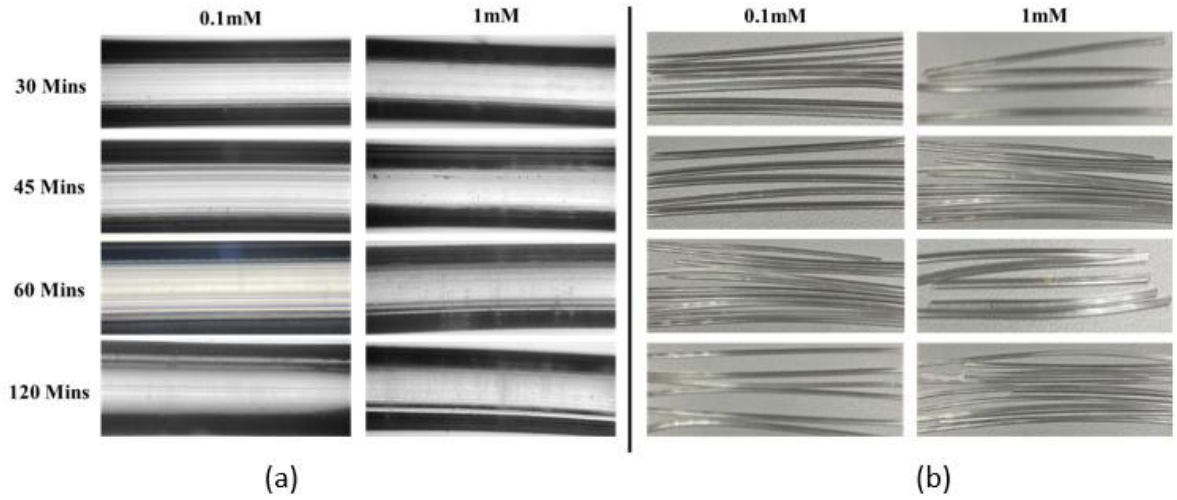

Fig 4 Macroscopic images of 3D-printed PETG optical fibers etched in 0.1 mM and 1 mM sodium hydroxide solutions for 30, 45, 60, and 120 minutes. No visible surface degradation such as cracks, pits, or delamination is observed. (b) Physical appearance of the etched fibers under ambient lighting conditions. All samples retained their original transparency and surface smoothness, with no signs of discoloration or optical defects across all etching durations and concentrations.

discoloration of the 3D-printed PETG fibers. This is particularly important, as previous studies have reported discoloration of PETG when immersed in various solvents, including distilled water, saline, and sugar solutions, with changes appearing as early as two weeks (Moreno Nieto et al., 2021). This preservation of optical clarity is critical for light-guiding applications, as surface irregularities or pigmentation could scatter or absorb light, thereby increasing optical loss. In this study, all etched samples retained their original optical appearance without compromising the visual or structural integrity of the PETG optical fibers.

Fourier Transform Spectra (FTIR)

Figure 5 presents the FTIR spectra of the etched 3D-printed PETG optical fibers. All characteristic peaks observed in the non-etched fibers remain present in the etched samples, indicating no chemical alteration due to the etching process. These peaks include bands at 831 cm^{-1} (=C–H), 1400–1409 cm^{-1} (CH_2 and C–O–C stretching), 1540 cm^{-1} (C=O), 1703 cm^{-1} (carbonyl stretching), and 2858 cm^{-1} (C–H stretching). These findings are consistent with spectral features reported in previous studies, where similar peaks were identified at 875, 1250–1050, 1450–1400, 1600–1500, 1750, and 2950–2850 cm^{-1} (Holcomb et al., 2022). No disappearance, shifting, or formation of new peaks was detected in the spectra of any etched samples, regardless of the NaOH concentration or immersion duration. This indicates that the mild etching conditions employed in this study do not induce any significant chemical changes to the PETG fiber structure. It is crucial that the chemical etching will not induce any structural degradation for PETG to be used in biomedical applications as degradation of petg could lead to increase toxicity of the polymer due to the release of the monomers (Martina et al., 2019).

In contrast, previous studies have reported a decrease in transmittance and an increase in absorbance of free hydroxyl and carboxyl groups indicative of PET hydrolysis when exposed to higher NaOH concentrations (5–10%) (Caparanga et al., 2009). Moreover, exposure to even stronger NaOH concentrations (5 M and 10 M) results in the emergence of broad O–H absorption bands around 3476 and 3434 cm^{-1}, respectively. These high concentrations also cause a reduction in the intensity and a shift of the strong C=O peak at 1716 cm^{-1}, indicating degradation of ester bonds (Maleque, 2023). Such degradation signatures were not detected in this study, reaffirming that the selected etching conditions (0.1–1.0 mM NaOH, up to 120 minutes) preserve the chemical integrity of the 3D-printed PETG fibers.

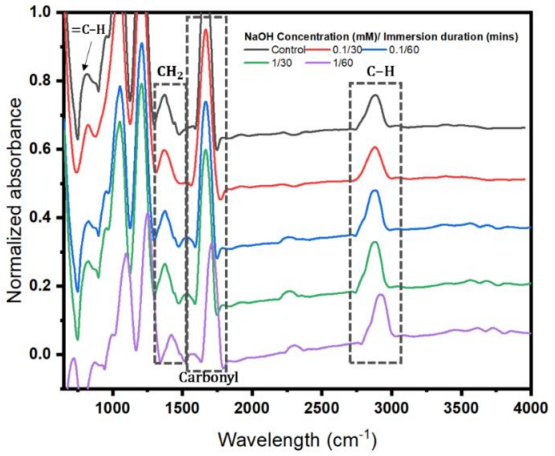

Fig. 5 Fourier-transform infrared (FTIR) spectra of the etched optical fibers etched with different NaOH concentrations and immersion durations. Dotted boxes highlight the key functional group regions such as =C–H, CH_2, C=O, and C–H stretches which show no disappearance or shifting across all samples, confirming the chemical stability of the PETG structure post-etching.

CONCLUSION

In conclusion, the chemical etching process used prior to hydrogel coating did not compromise the structural or optical integrity of the 3D-printed PETG optical fibers. A low optical

loss of 0.18 dB/cm was achieved after immersion in 1 mM sodium hydroxide for 2 hours, with no signs of surface degradation or chemical structure alteration observed through morphological and FTIR analyses. This stability is critical for maintaining biocompatibility, as degradation could increase the release of toxic by-products. The findings support the safe use of PETG in biomedical applications such as wound monitoring, phototherapy, and implantable devices. Future work should focus on detailed biocompatibility testing under physiological conditions and real-world assessments, including integration with hydrogel coatings, to further validate performance and safety in clinical environments.

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