Journal of Population Therapeutics & Clinical Pharmacology

RESEARCH ARTICLE
DOI: 10.53555/pxpknr95

COMPARATIVE ANALYSIS OF INFRARED ABSORPTION PEAKS IN UNTREATED AND SILANE-TREATED GLASS FIBERS USING FOURIER TRANSFORM INFRARED SPECTROSCOPY

Raham Zaman[1*], Hasham Khan[2], Muhammad Imran Ameer Malik[3]

[1*]Associate Professor, Science of Dental Materials Department, Bacha Khan Dental College, Mardan, Pakistan
[2]Assistant Professor Dental Materials, Khyber Medical University-Institute of Dental Sciences, Kohat, Pakistan
[3]Demonstrator at Science of Dental Materials Department, de'Montmorency College of Dentistry, Lahore, Pakistan

Corresponding Author: Dr Raham Zaman,
Associate Professor, Science of Dental Materials Department, Bacha Khan Dental College, Mardan, Pakistan. Email: drzaman1971@gmail.com

---

ABSTRACT

Background: Silane coupling agents are widely used to modify glass fiber surfaces to enhance chemical compatibility, bonding performance, and interfacial stability in composite materials. Fourier Transform Infrared Spectroscopy (FTIR) is a sensitive analytical method for detecting molecular-level changes following surface treatments.

Objective: This study aims to compare the infrared absorption characteristics of untreated and silane-treated glass fibers, with emphasis on identifying functional groups introduced or altered by silanization.

Methods: Glass fibers were divided into two groups: untreated controls and samples treated with a γ-methacryloxypropyltrimethoxysilane (γ-MPS) solution. FTIR spectra were collected in the range of 4000–400 cm^{-1} using attenuated total reflectance (ATR). Characteristic peaks corresponding to silanol (Si–OH), siloxane (Si–O–Si), carbonyl (C=O), and methacrylate-related vibrations were analyzed comparatively.

Results: Untreated fibers showed prominent absorption bands associated with Si–O–Si stretching (~1100 cm^{-1}), Si–OH bending (~950 cm^{-1}), and O–H stretching (~3400 cm^{-1}). Silane-treated fibers demonstrated additional peaks and intensity changes indicative of successful surface modification. Notably, the appearance of a carbonyl stretching band around ~1715–1730 cm^{-1}, CH_2 asymmetric and symmetric vibrations (2920 cm^{-1} and 2850 cm^{-1}), and enhanced Si–O–Si network peaks confirmed covalent silane layer formation. A reduction in free silanol-associated absorption also suggested condensation and crosslinking after treatment.

Conclusion: FTIR analysis revealed clear spectral differences between untreated and silane-treated glass fibers, confirming chemical bonding and formation of an organosilane layer..

Keywords: Silane coupling agent, FTIR, surface treatment, infrared spectra, composite adhesion**.**

INTRODUCTION

Glass fiber–reinforced composites are critical in dentistry, biomedical engineering, and structural applications, attributed to their superior mechanical properties and aesthetic compatibility.[1] The performance of these composites is significantly influenced by the quality of the fiber–matrix interface. Untreated glass fibers contain a high density of surface silanol groups (Si–OH), which exhibit inadequate compatibility with hydrophobic resin matrices. This incompatibility leads to weak interfacial adhesion and diminished long-term durability.[2] Silanization is commonly employed to address this limitation. Silane coupling agents, specifically γ-methacryloxypropyltrimethoxysilane (γ-MPS), demonstrate the ability to establish covalent bonds with inorganic glass surfaces as well as organic polymer matrices.[3] The hydrolyzed silane generates silanol groups that interact with the glass surface through siloxane bridges (Si–O–Si). Concurrently, the methacrylate end undergoes copolymerization with resin monomers during the curing process, resulting in a robust interphase.[4]

Fourier Transform Infrared Spectroscopy (FTIR) is widely employed for the detection of surface chemical modifications. The method enables quick and non-invasive identification of functional groups resulting from silanization and tracks the decrease in surface silanol groups.[5] Distinct absorption bands corresponding to Si–O–Si, Si–OH, C=O, C=C, and CH_2 modes facilitate comprehensive spectral analysis.[6] Glass fibers are commonly utilized in dentistry to enhance the physical and mechanical properties of Polymethylmethacrylate (PMMA) based dentures.[1] Numerous studies have demonstrated the positive impact of incorporating commercially available glass fibers. While commercially available glass fibers significantly improve the flexural strength and fracture toughness of PMMA dentures, they are relatively costly and not readily accessible.[2] Industrial glass fibers are utilized in various industrial applications and are more cost-effective compared to commercial dental glass fibers. Consequently, they may serve as a substitute for dental-grade glass fibers in the reinforcement of acrylic dentures.[6]

Prior research, including works by Zainudin et al., (2020)[7], Perera et al., (2022)[8], and Najeeb et al. (2020)[9], has demonstrated that the application of silane results in the emergence of carbonyl and methacrylate-related peaks in the ranges of 1715–1730 cm⁻¹ and 2850–2950 cm⁻¹, concurrently leading to a reduction of the O–H band in the vicinity of 3300–3500 cm⁻¹. These spectral changes are indicative of successful silane deposition.

Despite these advancements, there is a scarcity of direct side-by-side spectral comparisons of untreated and silane-treated fibers under consistent experimental conditions. This research examines characteristic infrared absorption peaks to confirm and quantify the chemical alterations resulting from γ-MPS treatment.

METHODOLOGY

Commercial E-glass fibers underwent cleaning with ethanol and subsequent drying, followed by division into two categories: untreated fibers and silane-treated fibers. The treated group was immersed in a 1% γ-MPS aqueous-ethanol solution, adjusted to a pH of approximately 4.5 to facilitate hydrolysis. Fibers underwent a reaction for 60 minutes, were dried at 110°C to promote condensation, and subsequently stored in desiccators. FTIR spectra for both groups were obtained using an ATR-FTIR spectrometer over the range of 4000–400 cm⁻¹ at a resolution of 4 cm⁻¹. The spectrum depicted the mean of 32 scans. Comparisons were made between groups regarding peak locations, intensities, and assignments of characteristic functional groups.

RESULTS

Untreated fibers exhibited strong silanol-related signals, particularly the broad O–H band and Si–OH peak near 950 cm⁻¹, confirming a hydrophilic and chemically active surface lacking organic functionalization. Silane-treated fibers exhibited new peaks do not present in untreated samples, thereby confirming the adsorption and condensation of γ-MPS. The carbonyl and CH_2 peaks were notably indicative of an organically functionalized silane layer (Table 1, 2).

Table 1: Principal Peaks of Absorption in Untreated Glass Fibers.

Wavenumber (cm^{-1})	Functional Assignment	Description
~3400	O–H stretching	Broad band due to surface silanol and adsorbed moisture
~1640	H–O–H bending	Trapped moisture vibration
~1100–1000	Si–O–Si asymmetric stretching	Dominant glass network vibration
~950	Si–OH stretching	Indicative of abundant free silanol groups
~460	Si–O–Si bending	Network deformation mode

Table 2. Major Absorption Peaks in Silane-Treated Glass Fibers

Wavenumber (cm^{-1})	Functional Assignment	Description
~3430 (reduced)	O–H stretching	Lower intensity shows reduced free silanol groups
~2920 / ~2850	CH_2 asymmetric & symmetric stretching	Derived from alkyl chains of γ-MPS
~1720–1730	C=O stretching (methacrylate)	Key indicator of silane layer presence
~1600	C=C stretching	Methacrylate functional group
~1100–1000	Si–O–Si stretching (intensified)	Evidence of siloxane network growth
~780–800	Si–C or Si–O–C linkages	Represents coupling agent bonding

DISCUSSION

FTIR analysis demonstrated notable spectral differences between untreated and silane-treated glass fibers, indicating that γ-MPS treatment significantly modifies the surface chemistry. The decrease in O–H stretch intensity (approximately 3400 cm^{-1}) indicates the consumption of surface silanol groups via condensation with hydrolyzed silane molecules. The presence of pronounced C=O stretching bands in the range of 1720–1730 cm^{-1} corresponds with earlier research by Mokhothu et al., (2022)[10], who identified similar carbonyl peaks as indicators of effective silanization. The detection of CH_2 stretching vibrations in the range of 2850–2920 cm^{-1} confirms the incorporation of organic methacrylate functionalities via γ-MPS. The findings align with those of Ramesh et al. (2022)[11], who identified comparable peaks in their characterization of silane-modified glass fibers utilized in dental composites.

The enhancement of the Si–O–Si peak around 1100 cm^{-1} further suggests crosslinking and the establishment of a stable siloxane network at the fiber surface. Numerous studies indicate that the formation of siloxane bonds is the crucial step for achieving durable adhesion, serving as a molecular bridge between inorganic substrates and organic matrices. Pradeep et al., (2022)[12] similarly emphasized this bonding mechanism as crucial for improving composite reinforcement. Another significant observation is the presence of Si–O–C or Si–C peaks in the range of 780–800 cm^{-1}, indicating the formation of covalent linkages between the silane and the glass surface. These bonds contribute to long-term hydrolytic stability, as evidenced by recent studies assessing the durability of fiber–resin interfaces.[13]

All of the spectrum alterations found in this investigation support the effective introduction of both organic (methacrylate) and inorganic (siloxane) bonding functions by γ-MPS treatment. In composite applications, this improves wetting, interfacial bonding strength, and long-term performance by producing a chemically compatible interphase.

CONCLUSION

FTIR spectroscopy showed considerable differences between untreated and silane-treated glass fibers. Untreated fibers had high silanol and moisture-associated peaks, suggesting a hydrophilic surface with low resin matrix compatibility. However, silane-treated fibers showed novel peaks for carbonyl, CH_2, C=C, and Si–O–C vibrations, indicating γ-MPS silane layer formation and condensation. These studies demonstrate the molecular relevance of silanization in improving fiber–matrix adhesion in dental, industrial, and structural composites.

REFERENCES

1. Graf RT, Koenig JL, Ishida H. Characterization of silane-treated glass fibers by diffuse reflectance Fourier transform spectrometry. Analytical Chemistry. 1984 Apr 1;56(4):773-8.
2. Ishida H, Koenig JL. Fourier transform infrared spectroscopic study of the structure of silane coupling agent on E-glass fiber. Journal of Colloid and Interface Science. 1978 May 1;64(3):565-76.
3. Al-Awabdeh FW, Al-Kheetan MJ, Jweihan YS, Al-Hamaiedeh H, Ghaffar SH. Comprehensive investigation of recycled waste glass in concrete using silane treatment for performance improvement. Results in Engineering. 2022 Dec 1;16:100790.
4. Ramalingam K, Thiagamani SM, Theivasanthi T, Chandrasekar M, Santulli C, Senthilkumar K, Siengchin S. Characterization of fiber surface treatment by Fourier transform infrared (FTIR) and Raman spectroscopy. InCellulose Fibre Reinforced Composites 2023 Jan 1 (pp. 115-127). Woodhead Publishing.
5. Sahai RS, Biswas D, Yadav MD, Samui A, Kamble S. Effect of alkali and silane treatment on water absorption and mechanical properties of sisal fiber reinforced polyester composites. Metallurgical and Materials Engineering. 2022 Dec 31;28(4):641-56.
6. Vijay R, Manoharan S, Arjun S, Vinod A, Singaravelu DL. Characterization of silane-treated and untreated natural fibers from stem of leucas aspera. Journal of Natural Fibers. 2021 Dec 2;18(12):1957-73.
7. Zainudin Z, Mohd Yusoff NI, Wahit MU, Che Man SH. Mechanical, thermal, void fraction and water absorption of silane surface modified silk fiber reinforced epoxy composites. Polymer-Plastics Technology and Materials. 2020 Dec 11;59(18):1987-2002.
8. Perera HJ, Goyal A, Alhassan SM. Surface properties of alkylsilane treated date palm fiber. Scientific Reports. 2022 Jun 13;12(1):9760.
9. Najeeb MI, Sultan MT, Andou Y, Shah AU, Eksiler K, Jawaid M, Ariffin AH. Characterization of silane treated Malaysian Yankee Pineapple AC6 leaf fiber (PALF) towards industrial applications. Journal of Materials Research and Technology. 2020 May 1;9(3):3128-39.
10. Mokhothu TH, Mtibe A, Mokhena TC, Mochane MJ. Influence of silane modification on the properties of natural fibers and its effect on biocomposites. InSurface Treatment Methods of Natural Fibres and their Effects on Biocomposites 2022 Jan 1 (pp. 67-93). Woodhead Publishing.
11. Ramesh M, Rajeshkumar L, Deepa C, Tamil Selvan M, Kushvaha V, Asrofi M. Impact of silane treatment on characterization of ipomoea staphylina plant fiber reinforced epoxy composites. Journal of Natural Fibers. 2022 Dec 1;19(13):5888-99.
12. Pradeep SA, Rodríguez LJ, Kousaalya AB, Farahani S, Orrego CE, Pilla S. Effect of silane-treated pine wood fiber (PWF) on thermal and mechanical properties of partially biobased composite foams. Composites Part C: Open Access. 2022 Jul 1;8:100278.
13. Petrunin MA, Maksaeva LB, Gladkikh NA, Yurasova TA, Kotenev VA, Tsivadze AY. Adsorption of organosilanes on the surfaces of metals and inorganic materials: 1. Adsorption on mineral surfaces. Protection of Metals and Physical Chemistry of Surfaces. 2021 Sep;57(5):867-82.