ORIGINAL ARTICLE

COMPARISON OF THE INFRA-RED ABSORPTION PEAKS OF UNTREATED AND SILANE TREATED GLASS FIBERS THROUGH FOURIER TRANSFORM INFRA-RED SPECTROSCOPY

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ABSTRACT

Fiber reinforced composites (FRCs) have a wide range of applicability in almost all fields of science. Recently, glass fibers have gained popularity in the field of dentistry owing to their excellent capability of bonding with PMMA denture base after silane treatment, biocompatibility, and esthetics. However, dental grade glass fibers are not only expensive, but they are also not readily available everywhere.

This study was aimed at evaluating the quality of coating of saline coupling agent that formed on industrial glass fibers, after they were treated with Trimethoxysilylpropyl methacrylate silane, and to assess the feasibility of using silane treated industrial glass fibers for dental usage, due to their low cost and abundant availability. This was done by comparing the infra-red (IR) absorption peaks of both types of woven E-glass fibers commercial (StickNET, Stick Tech, Finland and Industrial (Iqbal Sons, Karachi), by using Fourier Transform Infra-red (FTIR) spectroscopy.

The results indicated the presence of a polymerized layer of the silane coupling agent on both the commercial and industrial GFs. The absorption peaks on both types of glass fibers were identical, demonstrating that industrial glass fibers could be effectively used in replacement of commercial glass fibers, for the reinforcement of PMMA dentures.

Key Words: *Polymethylmethacrylate, mechanical properties, glass fibers.*

INTRODUCTION

Fiber reinforced composites (FRCs) have a wide range of applicability in almost all fields of science.[1] They are used in manufacturing of aircraft parts, automobiles, sports items and space equipment.[1] The FRCs have found their use in almost all of the fields of dentistry. In Prosthodontics they are being employed in the fabrication of denture bases[2,3], and fixed partial dentures.[4,5,6,7,8,9,10] They are also being tested for improvements in the mechanical properties of inlays and onlays[7,11,12], Periodontal splinting[2,7,8,9] and Orthodontic appliances.[4,5,6,7,8,9,12]

Glass fibers are frequently used in dentistry for improving the physical and mechanical properties of Polymethylmethacrylate (PMMA) based dentures.[13,14] Various studies have indicated the beneficial effect of adding commercially available glass fibers.[15,16,17]. Although these commercially available glass fibers provide excellent enhancement in the flexural strength and fracture toughness of PMMA dentures, they are quite expensive and they not easily available. The Industrial glass fibers are used in a variety of industrial applications, and are quite cost effective when compared with the commercial dental glass fibers. Therefore, they can be used as an alternative to the dental grade glass fibers for reinforcing acrylic dentures.[18]

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To ensure an optimal bond formation between the glass fibers and the polymer matrix, a silane coupling agent[19,20], usually Trimethoxysilylpropyl methacrylate (3-MPS) is coated over the surface of the commercial dental glass fibers.[19] In contrast, the industrial GFs are not pre-treated and they mustbe coated with a thin layer of 3-MPS before they can be used for reinforcement of PMMA dentures. The aim of this study was to compare the quality of a polymerized layer of silane coupling agent over the commercial (StickNET) and industrial woven E-glass fibers (Iqbal Sons, Karachi). This was done by comparing the infra-red (IR) absorption peaks of both types of glass fibers by using Fourier Transform Infra-red (FTIR) spectroscopy. The results indicated the presence of a polymerized layer of Trimethoxysilylpropyl methacrylate silane coupling agent on both the commercial and industrial GFs.

METHODOLOGY

The materials used in this study are given in Table 1.

FTIR spectroscopy (Thermo Nicolet 6700) was performed to detect the presence of a polymerized coating of MPS silane over the StickNET (Exp-I) and industrial (Exp-II) GFs which were treated with an 8% of Trimethoxysilylpropyl methacrylate (MPS) silane coupling agent used in this study. This was done by comparing the IR absorption spectra of the untreated Industrial GFs with both types of silane treated (Industrial and StickNET) glass fibers. The wave-number range was set between 4000-625 cm-1 having 256 scan cycles and a resolution of 8 cm-1.

Before running each sample a background spectrum was obtained without placing any specimen on the FTIR window. The spectra were recorded by total reflectance method using Attenuated Total Reflectance (ATR, eco ZnSe, Vmax in cm-1). The GF specimens were cut into small pieces and directly observed under the lens of the spectrophotometer.

RESULTS

The results of FTIR spectroscopy of the glass fibers are shown in Fig 1 and 2, where percent absorbance of the spectra is represented as a function of the wave number (cm-1). In case of the untreated Industrial glass fibers (Fig 1-A), the first peak of silica and metal (Si-O-M) can be identified at 900 cm-1 and the second peak of Si-O-Si can be seen at 1100 cm-1. At 1600 cm-1, and 1735 cm-1, very distinct third and fourth peaks of C=C linkages and C=O linkages can be observed while a fifth peak at 3750 cm-1 of free or isolated Si-OH bonds can be observed.

On the IR absorbance spectra of silane treated GFs, an Increase IR absorbance peak of the Si-OM and a decrease in the absorbance of Si-O-Si is visible in the Industrial and StickNET glass fibers (Fig 1 B and C) respectively, and indicates the chemical bonding of the silane coupling agent with the glass fibers. Combined absorption peaks of the untreated and silane treated industrial glass fibers and StickNET glass fibers is shown in Fig 2.

DISCUSSION

Trimethoxysilylpropyl methacrylate silane (MPS) is the most commonly used coupling agent in dentistry to promote chemical bond formation between the GFs and the PMMA resin. However, silanization of GFs is only effective when the silane coupling agent forms a polymerized layer over the GFs. In the current study, Fourier transform infra-red (FTIR) spectroscopy of Industrial (untreated and silane treated) and StickNET GFs was performed to observe and compare the IR absorption spectra of both GFs to assess and compare of a polymerized layer of silane coupling agent on the Industrial GFs which were treated with 8% MPS solution in 98% ethanol.

The absorption spectrum of the Industrial GFs in this study was quite like that of the StickNET GFs, which indicates towards the presence of a layer of silane coupling agent over the GFs. Si-O-Metal (900 cm-1), Si-OH (930 cm-1), Si-O-Si (1130 cm-1), C=C (1600 cm-1), C=O (1730 cm-1) and unbounded Si-OH (3750 cm-1), linkage peaks which are characteristic of bonds that are formed as a result of salinization were clearly visible on the spectra of both the silane treated GFs.

The peak of free hydroxyl (OH) groups at 3690 cm-1 which is significantly reduced in the both the silane treated specimens indicating the utilization of OH groups to form Si-O-Si linkages between the silane coupling agent and the glass fibers. In addition, a reduction in the peaks carbon- carbon double bond (C=C) and carbon- oxygen (C=O) bonds can be seen at 1635 and 1730 cm-1, respectively after the addition of MPS into the GFs.

In general, the IR absorption spectrum of the Industrial GFs was like that of the StickNET glass fibers that had been treated with a silane coupling agent by the manufacturer, which further corroborates the presence of a polymerized layer of MPS silane on the Industrial GFs. This indicates that both types of glass fibers possess a sufficiently thick coating of MPS Silane coupling agent, which can ensure an optimal bond formation between PMMA resin matrix and the GFs.

Glass fibers obtained from the industrial sources are cost-effective and easily available. In addition, a previous study by the author[3] indicated that industrial GFs that were separately coated with a silane coupling

TABLE 1: MATERIALS USED IN THIS STUDY

S. No.	Component Category	Type of Component / chemical nature	Brand name	Manufacturer
1	Glass fibers	Woven E-glass fibers	StickNet™	StickTech, Finland Iqbal Sons, Pakistan
2	Coupling Agent	98% (Trimethoxysilyl) propyl methacrylate silane (MPS)	Silane (A174)	Sigma Aldrich, USA

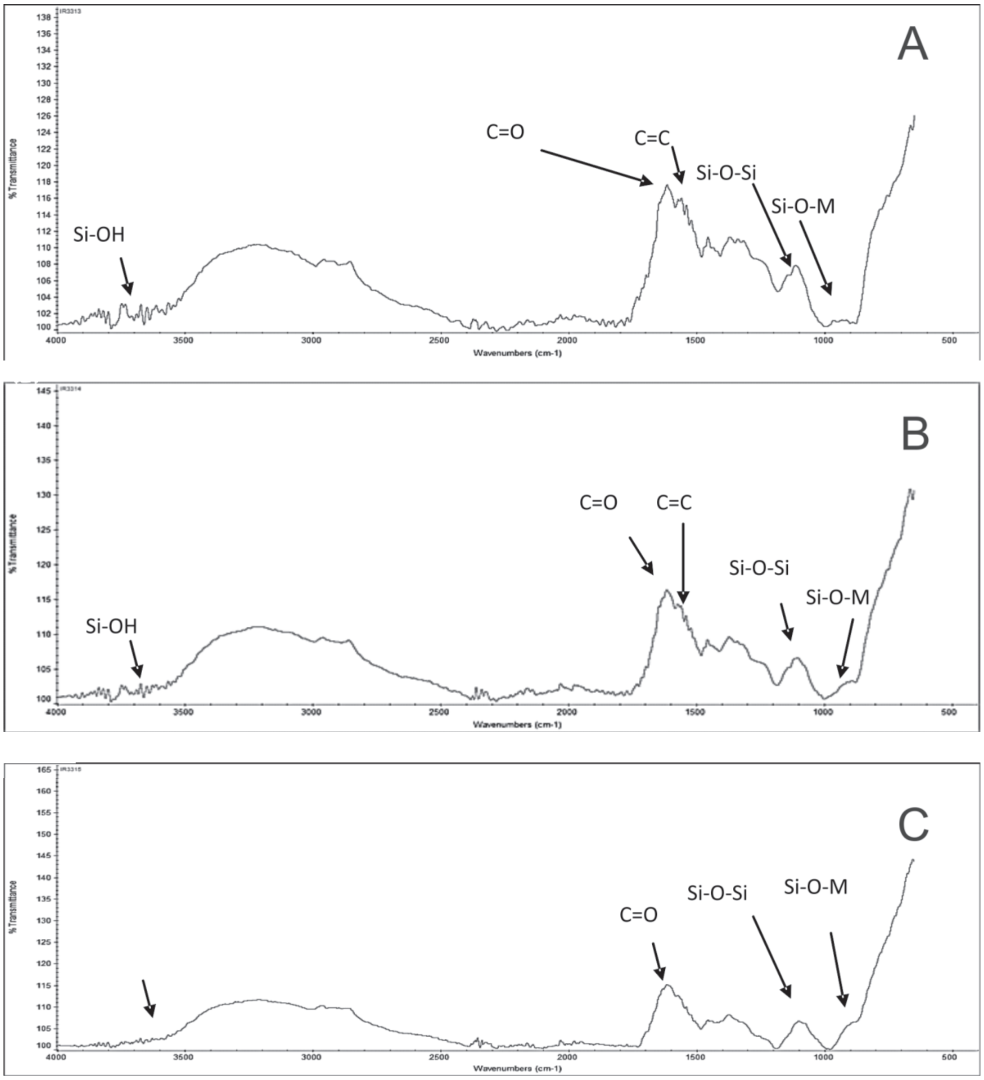

Fig 1: IR spectra of (A) untreated and (B) silane treated industrial glass fibers (C) commercial StickNET glass fibers.

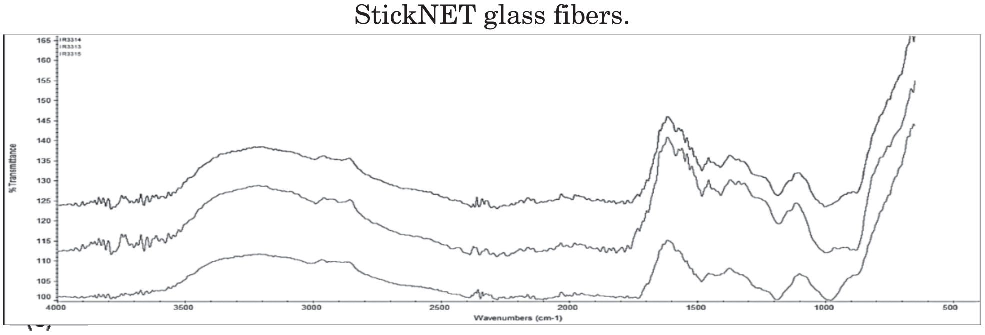

Fig 2: Combined IR spectra of (A) Silane treated industrial glass fibers (B) untreated industrial glass fibers (C) StickNET glass fibers

agent, possessed superior flexural strength in comparison to the unreinforced PMMA resins. Therefore, it can be safely said that industrial GFs can be used to reinforce PMMA dentures in place of expensive dental grade GFs.

CONCLUSIONS

From the results of this study, it can be concluded that:

- The coating of Silane coupling agent formed on the industrial E-glass fibers used in this study was similar to that seen in the commercial StickNET woven glass fibers.
- Industrial woven E-glass fibers can be effectively used to strengthen PMMA denture base materials after silane treatment.

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1 **Mehmood Asghar Bhatti:**	Conducted literature review, performed experimental work in the laboratory, prepared tables and figures, wrote the second and final draft after corrections made by supervisors.
2 **Shahab Ud Din:**	Supervisor, helped in developing evaluating the experiments and statistical analysis, refined the first draft written by the first author, proof reading of the final draft for publication.
3 **Muhammad Kaleem:**	Acted as co-supervisor, helped in developing methods & in experimental work, read also the final draft.
4 **Muhammad Hassan:**	Helped in experiments, in data collection, refined first draft & helped use of SPSS for data analysis, refined the first draft written by first author, helped in use of SPSS for data analysis.
5 **Bushra Naureen:**	Helped in developing the methodology of the experiment, in interpretation of the results & Performed identification of bonds from their respective absorption peaks.