Acta Odontologica Scandinavica. 2014; 72: 509–515

ORIGINAL ARTICLE

Adhesion between glass fiber posts and resin cement: evaluation of bond strength after various pre-treatments

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Abstract

Purpose. To evaluate surface roughness and bond strength of glass fiber posts to a resin cement after various surface treatments. ***Materials and methods.*** Sixty individually formed glass fiber posts with a diameter of 1.5 mm and a length of 20 mm were used for this study. They were randomly assigned to six groups of pre-treatment (n = 10/group): Group C, untreated (control); Group SB, sandblasted; Group SC, silica coated; Group HF, hydrofluoric acid-etched; Group N, Nd:YAG laser irradiated; Group E, Er:YAG laser irradiated. Surface roughness of the posts was measured before and after pre-treatment. The posts were then bonded to resin cement and tensile bond strengths were determined in a universal testing machine. For statistical analysis, two-way ANOVA and post-hoc comparison tests ($\alpha = 0.05$) were performed. ***Results.*** The highest bond strength value was observed in group HF, followed by group SC. There was a statistically significant difference in bond strength between group C and groups HF, SC and E ($p < 0.001$, $p = 0.002$ and $p = 0.041$, respectively). Posts of group SB and group N showed the highest surface roughness. ***Conclusions.*** The findings of the present study reveal that hydrofluoric acid-etching, silica coating and Er:YAG laser irradiation provided a significant increase in bond strength between glass fiber posts and resin cement.

Key Words: *acid-etch, bond strength, glass fiber post, laser, sandblasting, silica coating*

Introduction

The primary purpose of a post is to retain the coronal restoration in an endodontically treated tooth that has suffered an extensive loss of crown structure because of decay, excessive wear or prior restoration [1–3]. The restoration is supported by a cast or pre-fabricated post and core system [4]. Pre-fabricated post systems have become more popular because they can provide satisfactory results while saving time and reducing costs [5,6]. With the increasing demands for esthetic restorations, pre-fabricated fiber posts including glass fiber, quartz fiber, carbon and polyethylene fiber, which were introduced in the 1990s, are used as an alternative to metal posts to restore endodontically-treated teeth [7–10]. Having an elastic modulus similar to dentin (≈20 GPa), fiber posts provided a better distribution of loads to the teeth than metal posts or conventional cast posts and induced a low rate of non-retrievable root fracture, the most serious type of failure [1,4–16].

Glass-fiber posts are composed of glass fibers, inorganic filler and polymer matrix, commonly an epoxy resin or other resin polymers which have a high degree of conversion and highly cross-linked structures [5,15,16]. Glass-fiber posts can be made of different types of glass. Electrical glass which consists of a mixture of SiO_2, CaO, B_2O_3, Al_2O_3 and some other oxides of alkali metals, is the most commonly used glass type and also the most economical glass fiber for composite resins [17]. Glass fibers have elasticity, high tensile strength, low electrical conductivity, resistance to solubility or corrosion and resistance to biochemical degradation. Furthermore, it is easy to remove them into the root canal [15]. Furthermore, Goldberg and Burstone [18] reported that glass fiber post systems strengthened the

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(Received 24 September 2013; accepted 3 December 2013)

ISSN 0001-6357 print/ISSN 1502-3850 online
DOI: 10.3109/00016357.2013.875586

structure of the post without compromising the modulus of elasticity.

Design, length, diameter and the surface treatment of the post are factors that affect retention of a post [5,19]. Failure of glass fiber-reinforced posts often occurs because of de-bonding at the post–adhesive interface as a result of bond deficiencies [3,6,12,16]. Therefore, in order to maximize resin bonding to fiber posts, many surface pre-treatment procedures including mechanical or chemical treatments of the post surface have been investigated [6,7,10,16]. These procedures fall into three categories: (1) treatments that result in chemical bonding between a composite and post (coating with priming solutions); (2) treatments that intend to roughen the surface (sandblasting and etching); or (3) combine micromechanical and chemical components either by using the two above-mentioned methods or a unique system (such as Co-Jet) [15,16,19,20].

Silane application would promote adhesion by increasing the post surface wettability, as well as by chemically bridging methacrylate groups of the resin and hydroxyl groups of glass fibers [1,21]. On the other hand, sandblasting procedure involves spraying a stream of aluminum oxide (Al_2O_3) particles with different particle sizes ranging from 30–250 μm against the material surface intended for making materials more bondable under high pressure [22]. It is commonly employed in ceramic procedures and for pre-treatment of the metal surface [14].

Etching with hydrofluoric acid (HF) is intended to create a roughening of the surface, which allows for micromechanical interlocking with the resinous cement. The acid effect was time-dependent and influenced by the post composition such as type of matrix and fibers [19,20].

Silica coating or tribochemical coating with the Co-Jet system (Co-Jet, 3M ESPE, St Paul, MN) provides ultrafine mechanical retention by relying on the use of Al_2O_3 particles modified by silica with 30 μm grain size. As a result, a silicate layer is welded onto the post surface by high spot heat produced by blasting pressure which results in embedding of silica particles on the surface rendering the surface chemically more reactive to resin via silane [19,20,23].

In dentistry, laser technology is one of the last developments that may have an impact on the alteration of the materials surface for improving roughness and bond strength [24–28]. Recently, lasers have been shown to be relatively safe and proposed for different dental applications such as roughening ceramics [25,26], acrylic resins [27,28], etching metals before the application of porcelain [29], reducing tooth sensitivity [30], removing caries [31] and bleaching [32]. Among the various laser types employed in dentistry, the neodymium:yttrium-aluminum-garnet (Nd:YAG) laser operates at a wavelength of 1.064 μm in a high-intensity pulsed waveform, whereas erbium: yttrium-aluminum-garnet (Er:YAG) laser operates at a wavelength of 2.94 μm and in a pulsed waveform [27]. Moreover, Er:YAG laser's strong reputation results from the fact that its wavelength emission coincides with the main absorption peaks of water (≈3.0 μm) [33]. These two types of laser were the most highly recommended lasers in dentistry to carry out roughening of materials, due to their high power.

There is little information in the existing literature on the bonding capability of pre-treated glass fiber posts to resin cement. Therefore, the purpose of this *in vitro* study was to investigate effects of different surface treatments including sandblasting, HF, silica coating, Nd:YAG and Er:YAG laser treatments on the strength of the bond between glass fiber posts and resin cement and surface roughness of the posts. It was hypothesized that surface modifications would not significantly affect the bond strength between the glass fiber posts and resin cement.

Materials and methods

The study protocol was approved by the local Ethics Committee in Research of the local institution (Cumhuriyet University), protocol 2012-01/09.

Translucent and individually formed glass fiber posts (Everstick; StickTech, Turku, Finland), with a diameter of 1.5 mm, were used for this study. Sixty glass fiber posts with a length of 20 mm were prepared and light-cured for 60 s (550 mW/cm^2, Hilux 550; Hilux, Ankara, Turkey). Materials and their compositions used in this study are presented in Table I. Posts were then randomly divided into six experimental groups (n = 10), according to the surface treatments performed: control (C), sandblasted (SB), silica coated (SC), hydrofluoric acid-etched (HF), Nd:YAG laser irradiated (N) and Er:YAG laser irradiated (E).

Prior to surface treatments, the surface roughness (Ra) values were measured with a surface roughness profilometer (Mitutoyo surftest SJ-401; Mitutoyo Corporation, Kanagawa, Japan) with a diamond stylus (tip radius 5 μm). A reading was obtained from the needle passing across 0.8 mm length at 1 mm/s to the nearest 0.01 μm. This procedure was repeated one more time in the same position for a total of two readings. Thus, two readings, each consisting of two lengths of 0.8 mm, were made at each position, resulting in a total reading length of 1.6 mm. A final Ra average was calculated and the means of individual specimens were averaged.

- Group C—untreated (control): This group served as the control group, so no treatment was applied to the post surfaces in this group.
- Group SB—sandblasted: Bonding surfaces of post specimens were sandblasted (Ney, Blastmate II, Yucaipa, CA) with 110 μm Al_2O_3 for 10 s. The air

Table I. Materials and their compositions used in this study.

Materials	Type	Composition	Manufacturer
Everstick post	Unidirectional E-glass	48% glass fiber, semi-IPN of PMMA, Bis-GMA, TEGDMA	StickTech, Turku, Finland
DUO-LINK	Dual-cured composite luting cement	Base: Bis-GMA, TEGDMA, UDMA, glass filler Catalyst: Bis-GMA, TEGDMA, glass fiber	Bisco, Inc., Schaumburg, IL
Rocatec Pre	Al_2O_3 particles	Al_2O_3 particles size 110 μm	3M ESPE, St Paul, MN
CoJetSand	Tribochemical silica-coating particles	Silica-modified Al_2O_3 particles of 30 μm	3M ESPE, St Paul, MN
Ultradent	Porcelain acid gel	9.5% hydrofluoric acid gel	Ultradent Products, Inc., UT

Semi-IPN, semi-interpenetrating polymer network; PMMA, polymethylmethacrylate; Bis-GMA, bisphenol glycidyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate; Al_2O_3, Aluminium oxide.

pressure for sandblasting was maintained at 2 bars. Specimens were perpendicularly mounted in a special holder at a distance of 10 mm between the surface of the specimen and the blasting tip. Then, the specimens were rinsed under running water and then dried with oil-free compressed air to remove the remnants for 10 s.

- Group SC—Silica coated: The posts were abraded with an intra-oral airborne-particle-abrasion device (Co-Jet, 3M ESPE, St Paul, MN) using 30 μm silica-modified Al_2O_3 particles (CoJet Sand) while rotating the post manually for 10 s. The abrasive was applied perpendicular to the specimens' surface at 2 bars pressure from a distance of 10 mm.
- Group HF—Hydrofluoric acid-etched: The posts surfaces were etched with 9.5% hydrofluoric acid (Ultradent Products, Inc., South Jordan, UT) for 20 s.
- Group N—Nd:YAG laser irradiated: Bonding surfaces of post specimens were irradiated by Nd:YAG laser (Smarty A10, Deka Laser, Florence, Italy) with contact, no distance. Laser energy was delivered in pulse mode with a 300 mm diameter laser optical fiber for 20 s. The laser parameters used were 100 mJ (pulse energy), 10 Hz (repetition rate), 1 W (output power), 300 μs (pulse duration) and 141.54 J/cm^2 (energy density). Furthermore, air cooling was used during laser irradiating of the specimens.
- Group E—Er:YAG laser irradiated: The bonding surfaces of the post specimens were irradiated with an Er:YAG laser (Smart 2940D Plus, Deka Laser, Firenze, Italy). Laser energy was delivered in pulse mode by a 4 mm diameter titanium articulated arm transmission system with a repetition rate of 10 Hz, energy of 150 mJ, output power of 1.5 W, energy density of 119.42 J/cm^2 and a pulse duration of 700 μs for 20 s. The distance of application was 10 mm. In addition, water irrigation was used during the lasing of the post specimens.

Representative post specimens were examined under a scanning electron microscope (SEM, LEO 440, Zeiss, Oberkochen, Germany). They were photographed at 1000× magnification. SEM analysis was performed with secondary electron (SE) mode under 20 kV accelerating voltage in vacuum (3×10^{-4} Pa) and 80 μA beam current.

Following surface treatments, surface roughness of the posts were measured as described before. For preparing tensile test specimens, a plastic tube (10 mm height) was used. It has a small central pit in order to obtain a standardized central position of the post. Prior to cementation procedure, an adhesive resin (ONE-STEP PLUS, Bisco, Inc., Schaumburg, IL) was applied to the posts' surfaces and light cured (Hilux 550; Hilux) for 10 s according to the manufacturer's instructions. Resin cement is already in a plastic syringe and includes mixing tips, thus equal amounts of paste A and B were dispensed. The resin cement material (DUO-LINK, Bisco, Inc.) was injected into the plastic tube; posts were placed into it and light-cured for 40 s according to the manufacturer's instructions, using a halogen light curing unit (Hilux 550; Hilux). Additional 40 s irradiations were performed from each side of the cylinder prior to the removal of the specimens into the plastic tube to ensure optimal polymerization of the resin material. After removing cemented posts from the plastic tube, retention holes were prepared on the surfaces of the remaining parts of the posts (the portion of the post that extended from the cement) with a round diamond bur in order to improve retention of the acrylic resin. Posts specimens were then placed into a silicone mold and an autopolymerizing acrylic resin material was applied into the mold.

After the specimens were stored in distilled water at 37°C for 7 days in a water bath machine (BM 402, Nüve, Ankara, Turkey), they were attached to a custom jig of a universal testing machine (Lloyd LF Plus, Ametek Inc, Lloyd Instruments, Leicester, UK) and subjected to a tensile load parallel to the long axis of the posts at a crosshead speed of 1 mm/min until failure occurred. Furthermore, the fractured specimens were examined under a stereomicroscope (SMZ 800) at 40× magnification to evaluate the fracture pattern and modes of failure were categorized into one of three categories: *adhesive failure* refers to total separation at the interface between the post and resin cement; *cohesive failure* refers to fracture within the post or resin cement; *mixed failure* refers to both. All fracture observations were conducted by one person. The data were analyzed using a two-way ANOVA test. In addition, *post-hoc* comparison tests were performed at a 0.05 significance level.

Results

Two-way ANOVA test results for tensile bond strength and surface roughness measurements of the groups are summarized in Table II. Analysis of the data revealed significant differences among the different surface treatment groups ($F = 10.424$ and $p < 0.001$). The highest mean bond strength value was observed in group HF and followed by group SC and no significant difference was found between groups HF and SC ($p = 0.744$). Tukey's HSD test also showed that no significant difference was found between groups HF and E and groups SC and E ($p = 0.165$ and $p = 0.896$, respectively). Moreover, a statistically significant difference in tensile bond strength was found between group C and groups HF, SC and E ($p < 0.001$, $p = 0.002$ and $p = 0.041$, respectively). The lowest mean bond strength value was observed in group N. In addition, there was no statistical difference between group C and groups SB and N ($p = 0.085$ and $p = 0.999$, respectively). Laser groups (N and E) presented significantly different bond strength ($p = 0.016$). The bond strength of group SB was significantly higher than that of group N ($p = 0.035$).

Modes of failure are also evaluated. The analysis of failure after the tensile bond strength test revealed that the adhesive failure mode was predominantly observed in groups of C and N. However, other groups predominantly exhibited cohesive failure modes between resin and post interface.

Table II. Mean tensile bond strength (N) and surface roughness (Ra) and SD values obtained for each tested group.

Groups	Bond strength	Roughness
Group C	546.6^{ab} (50.54)	0.98^{A} (0.53)
Group SB	619.2^{bc} (50.77)	4.24^{B} (0.97)
Group SC	655.2b^{c} (58.24)	2.27^{C} (0.62)
Group HF	691.6^{c} (55.88)	1.14^{A} (0.44)
Group N	537^{a} (83.74)	4.15^{B} (1.11)
Group E	627.1^{c} (49.97)	3.62^{B} (0.84)

For bond strength: values with small letters indicate no statistically significant difference ($p > 0.05$).
For surface roughness: values with capital letters indicate no statistically significant difference ($p > 0.05$).

When evaluating roughness results ($F = 34.508$), the highest mean value was observed in group SB and followed by group N. Tukey's HSD test showed that no significant difference in surface roughness was found between group C and group HF ($p = 0.998$); however, differences between group C and other groups (SB, SC, N and E) were statistically significant ($p < 0.001$, $p = 0.008$, $p < 0.001$ and $p < 0.001$, respectively). Furthermore, the surface roughness of group SB was significantly higher than those of groups SC and HF ($p < 0.001$), contrary to groups N and E ($p = 1$, and $p = 0.512$). Differences between group SC and all other groups were found to be statistically significant ($p < 0.05$). On the other hand, there was no significant difference in roughness between laser groups, group N and group E ($p = 0.675$).

SEM micrographs of the representative specimens of the investigated pre-treatment procedures are presented in Figures 1A–F. It can be seen that untreated, silica-coated and HF acid-etched posts exhibited more flat surface than Nd:YAG laser irradiated post. Moreover, surfaces of the sandblasted and Er:YAG laser irradiated posts showed distinctive irregularities. There was an important difference between these surface treatments in SEM evaluations. Fiber ruptures were seen in the sandblasted post, whereas Er:YAG laser irradiation resulted in separation among fibers.

Discussion

The results obtained in this study clearly demonstrate that post surface treatments including silica coating, HF acid-etching and Er:YAG laser irradiation significantly affected the tensile bond strength of glass fiber posts to resin cement. Therefore, the null hypothesis was rejected. Although, sandblasted and Nd:YAG laser irradiated posts had the highest mean roughness value, they were not presented significant higher tensile bond strength value than that of control group value.

It was reported that glass fiber posts submitted to sandblasting were rougher and appeared to provide an increased surface area, which could suggest improvement in mechanical interlocking for the resin cement [15]. However, this procedure did not increase bond strength values of the posts. Thus, these results were consistent with those of the present study. Nevertheless, the highest bond strength value on sandblasted posts was recorded and, according to this study, SEM evaluation supported bond strength data revealing a more retentive surface created by sandblasting [14]. In accordance with these findings, it was reported that sandblasting of the surface of glass fiber posts improved retention and provided a rougher surface area [5]. Moreover, it was demonstrated that sandblasted posts exhibited higher bond strength valued than phosphoric acid-etched posts [12]. The results of the present study contradicted those of Radovic et al. [14], Balbosh and Kern [5] and Albashaireh et al. [12]. Furthermore, it was stated that surface treatments with 9.6% HF acid gel for 60 s and sandblasting did not improve the strength of the bond between individually formed glass fiber posts and resin-core materials [7]. In agreement with the present study, it was presented that HF acid application significantly enhanced the interfacial bond strength between fiber posts and core materials [10]. This study also showed that HF acid alters the post structure more radically. This finding was understandable, because; not only was a low percentage of HF (4%) applied on the surface of the posts but also a long application time (60 s) was preferred. Hence, 9.5% HF acid gel was applied on the posts' surface for 20 s in the present study.

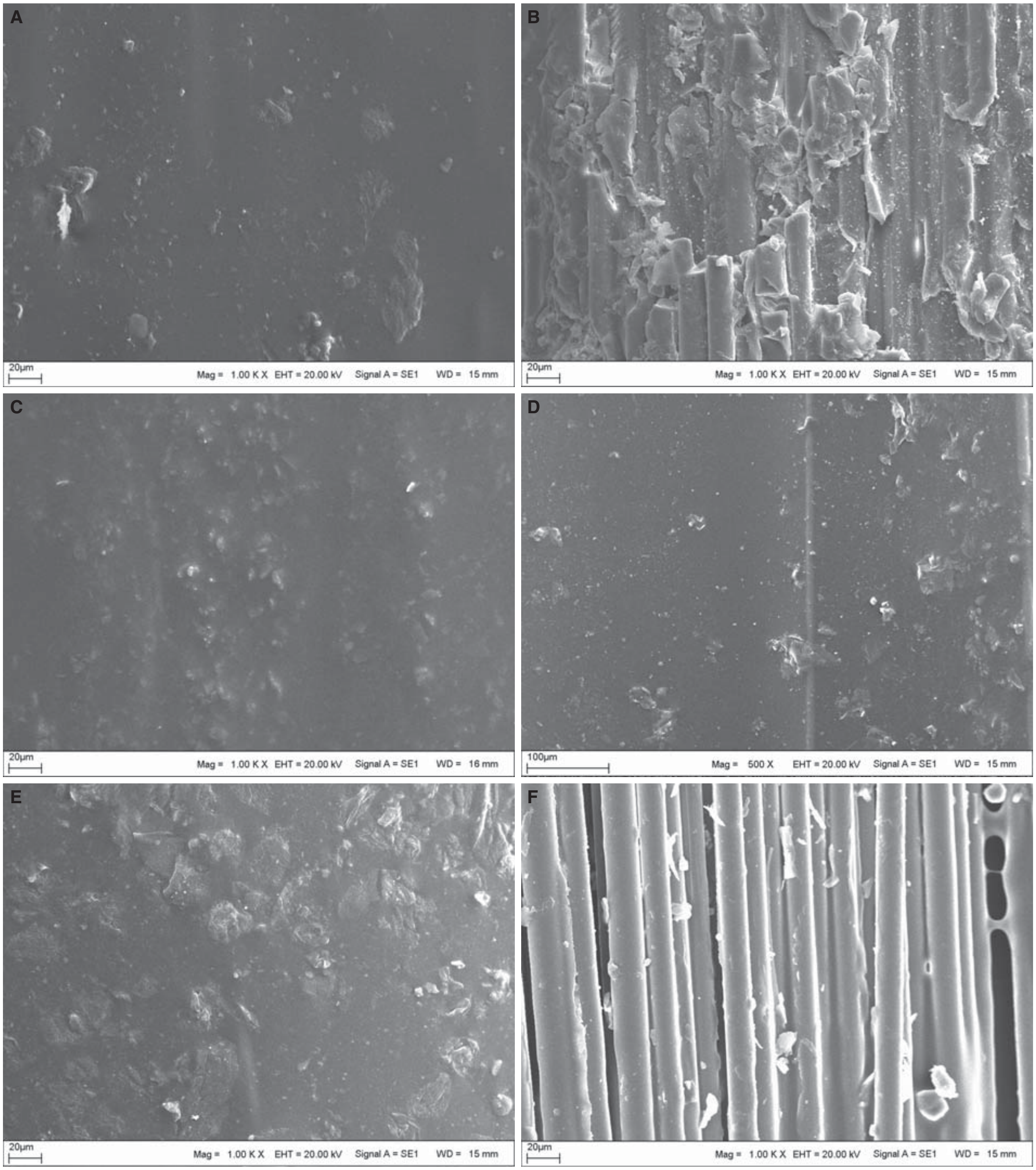

Figure 1. Representative SEM micrographs of the glass fiber posts' surfaces (original magnification: 1000×). (A) Untreated post. (B) Sandblasted post. (C) Silica-coated post. (D) HF acid-etched post. € Nd:YAG laser irradiated post. (F) Er:YAG laser-irradiated post.

On the other hand, it was demonstrated that none of the surface treatments including sandblasting, HF acid-etching and silica coating had an effect on the bonding to glass fiber posts [34]. In the present study, silica coating was found to be effective for improving bond strength of the glass fiber posts. However, this was not higher than HF acid-etching. Thus, the results of the present study were not in accordance with those of Sahafi et al. [34].

The silicate layer is welded onto the post surface by high spot heat produced by blasting pressure in a process called tribochemical coating. These procedures are followed by silanization of the pre-treated fiber post surface, thus combining chemical and micromechanical retention [19]. To improve the bond strengths between adhesive luting cements and glass fiber posts, silanization of the post surface has been suggested but revealed contradictory effects [35,36]. In the present study, silica coating was found to be an effective method for improving bond strength of the glass fiber post. Inconsistent with the present study, silanization proved to be significant for improving bond strengths of glass fiber posts [35], nevertheless, it was advocated that effects of silanization appeared to be clinically negligible [35,36]. Furthermore, the type of fiber post [35] and resin cement [36] revealed a significant influence on bond strengths.

tIn addition, some of the researchers [5,12] sandblasted glass fiber posts with 50 μm Al_2O_3 particles at 2.5 bar pressure for only 5 s from a distance of 30 mm, whereas different application procedures were seen in other studies [7,12,14,15] (at 2 bar for 10 s from a distance of 10 mm or at 2.8 bar for 5 s from a distance of 10 mm or 110 μm Al_2O_3 particles from a distance of 10 mm at 2.8 bar for 5 s). According to the pilot study, it was found that sandblasting of glass fiber posts with 110 μm Al_2O_3 particles at higher than 2 bar from a distance of 10 mm produced pronounced dimensional changes in the form of the posts. Therefore, in this study the surfaces of the posts were sandblasted with 110 μm Al_2O_3 particles at 2 bar pressure for 10 s from a distance of 10 mm. This regimen did not produce visible changes in the form of the posts.

It was reported that airborne particle abrasion or Er:YAG laser irradiation applied on quartz fiber posts did not affect the push-out bond strengths relative to the root surfaces [37]. Contrary to the results of this report, different lasers exhibited different results in the present study. Er:YAG laser irradiated posts demonstrated significantly higher bond strength than Nd:YAG laser irradiated posts. It can be explained that Nd:YAG laser application resulted in larger pits than Er:YAG laser application on the surfaces of the posts. Most reports [4,38] evaluated adhesion between posts and laser irradiated root canal dentin in the literature. Both for Nd:YAG and Er:YAG laser applications, many different experimental set-ups have been used in the literature and application of lasers in different parameters such as energy, output power and pulse duration can affect the results of the studies. Hence, to determine the laser parameters used in this study, a pilot study was carried out. Based on those results and a previous study [24], 100 mJ, 10 Hz and 1 W for Nd:YAG laser, and 150 mJ, 10 Hz and 1.5 W for Er:YAG laser were selected. In addition, the distance of the laser application was selected based on the previous studies [24–28].

On failure after tensile bond strength testing, different failure types were observed among the groups and there was a correlation between tensile bond strength and failure types. Group SC and HF which had the highest bond strength values presented cohesive failure modes (80% and 70%, respectively).

According to SEM evaluations, it could be expected that HF and SC groups of specimens presented similar bond strength values with a control group of specimens. Nevertheless, the highest bond strength values were seen in these groups of specimens. Thus, no correlation was found between bond strength and surface roughness values. Etching with hydrofluoric acid is intended to create a roughening of the surface, which allows for micromechanical interlocking and the effect of it has been influenced by the post composition [19]. Moreover, silica coating is based on a combination of chemical and micromechanical retention. Therefore, both mechanical and chemical bond should be evaluated between fiber post and resin cement. On the other hand, Er:YAG laser irradiation significantly affected the tensile bond strength of glass fiber posts to resin cement. It could be explained that resin cement could penetrate into the cavities which are produced by separation of fibers. However, these fiber separations could impair structure of the post. Therefore, future investigations could focus on fracture strength of the fiber posts after Er:YAG laser irradiation.

Conclusion

Within the limitations of this *in vitro* study the following conclusions were drawn:

(1) Surface treatments with hydrofluoric acid, silica coating and Er:YAG laser irradiation were found to be effective methods for improving the bonding of glass fiber posts to resin cement.
(2) No correlation was found between bond strength and surface roughness.

Acknowledgements

This investigation was supported in part by the Cumhuriyet University Scientific Research Project. This study was presented in a poster at the annual meeting of the 19th Aegean Region Chambers of Dentists International Scientific Congress and Exhibition, 26–28 April, 2012, Fethiye, Turkey.

Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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