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Effects of Heat Treating Silane and Different Etching Techniques on Glass Fiber Post Push-out Bond Strength

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Clinical Relevance

Surface treatment enhances bond strength of a fiber post. Heat treating silane improves its efficiency.

SUMMARY

The aims of this study were to compare two pretreatment methods of a fiber post and to evaluate the effect of heat treatment to applied silane on the push-out bond strength for different levels of root. In this *in vitro* study, 40 glass fiber posts were divided into five groups (n=8) according to the kind of surface treatment applied. They were then inserted into extracted and endodontically treated human canines using a self-etch resin cement (Panavia F2.0, Kuraray, Japan). Group HF+S = hydrofluoric acid (HF) etching and silane (S) application; group HF+S+WP = HF etching and heat-treated silane application and warmed posts (WP); group H_2O_2+S = hydrogen peroxide etching and silane application; group H_2O_2+S+WP = hydrogen peroxide and heat-treated-silane application and warmed post; and group C, the control group, received no pretreatment. After completion of thermal cycling (1000 cycles, 5-55°C), all specimens were cut horizontally to obtain three sections. Each section was subjected to a push-out test, and the test results were analyzed using two-way analysis of variance, post-hoc Tukey honestly significant difference test, and a paired sample *t*-test (α=0.05). It was found that bond strength was not statistically influenced by the kind of etching material used (p=0.224), but was significantly affected by heat treatment of applied silane ($p<0.001$). The interaction between these two factors was not statistically significant (p=0.142). Group HF+S+WP showed the highest bond strength (12.56±1.73 MPa) ($p<0.05$). Scanning electron microscopy revealed the effect of the different treatments

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DOI: 10.2341/11-486-L

on the surface characteristics of posts. In the four pretreated groups, the bond strength decreased significantly from the coronal to the apical root canal sections ($p \leq 0.05$). The results of this study show that the use of heat-treated silane significantly enhances the push-out bond strength of the fiber posts to root. HF acid etching with heat-treated silane application led to the highest bond strength.

INTRODUCTION

Endodontically treated teeth may weaken due to caries, fracture, previous restorations, and access cavity preparation. Casting posts are frequently used to restore such teeth that have an insufficient coronal structure; the aim is to provide adequate retention for future prosthetic restorations.[1-3] Traditional metallic cast posts are gradually being replaced by fiber posts.[4,5] The latter have several mechanical and clinical advantages, such as esthetic appeal, corrosion resistance, easier removal for endodontic retreatment, faster insertion, and decreased probability of catastrophic root fracture.[3,5,6] The last of these advantages is the most important and is mainly attributed to the elastic modulus of fiber posts, which is closer to the modulus of dentin and improves the distribution of occlusal stresses to the root dentin. Failure of fiber posts and core restorations usually occurs from debonding or, less often, from fracture of the fiber post.[2,4,5] In the case of inadequate bond strength, debonding occurs between the fiber post/resin and/or the resin/root canal dentin interfaces.[2,7] The matrix of fiber posts is usually composed of highly cross-linked epoxy resin with a high degree of conversion. Silanes do not generally bond well with the epoxy matrix. A chemical bond via application of silane may be obtained only with the exposed glass fibers of the post.[2] Several factors are known to affect the retention of posts, such as post length and design, type of luting agent, cementation procedure, and preparation, shape, and condition of the post space.[8-10]

Various pretreatment methods involving the use of mechanical or chemical agents have recently been proposed for improving resin bonding to fiber posts.[11-13] Although several researchers propose the use of silane coupling agents to form a strong bond between the composite resin and the fiber post, there are several different views about the efficiency of post silanization. Further, silane application is known to be a technique-sensitive step. Numerous factors influence the effectiveness of silane, such as its composition (pH, presence of solvent, molecular size) and the application method used. The chemical reaction between silane and an inorganic surface may be enhanced and catalyzed by acid treatment of the fiber-post surface or by silane heating,[14] particularly in the case of two-component silane solutions, which have been shown to be more sensitive to heating.[15]

Surface etching of fiber posts using chemical agents such as hydrofluoric acid (HF), hydrogen peroxide (H_2O_2), hydrochloric acid, and potassium permanganate produces a rough surface and therefore increases mechanical retention between the post and the resin luting agent. Although the use of HF achieves this purpose, it may damage glass fibers and affect the integrity of the post. H_2O_2 etching is a considerably milder technique, in addition to being simple, effective, and clinically feasible for enhancing bond strength.[2,16]

The effect of H_2O_2 on bond strength has been evaluated through microtensile tests.[16,17] However, because a push-out test is more reliable than a microtensile test,[18] in this study the former was used to evaluate bond strength. In studies thus far, warm air has been used for heat treatment of a silanated fiber post surface. Heat treatment of silane with warm water may result in the formation of a thin layer of silane and may enhance bond strength.

The present *in vitro* study was performed to compare the effects of two kinds of chemical etching pretreatments of the fiber-post surface on the push-out bond strength and to determine whether the heat treatment of the applied silane solution with warm water could increase the bond strength to exceed that resulting from a room-temperature air-drying procedure. The null hypotheses tested in this study were as follows: 1) heat treatment of silane solution does not affect the push-out bond strength of fiber posts, 2) there is no difference in bond strength between different etching techniques, and 3) there is no difference in bond strength at different root levels.

METHODS AND MATERIALS

Specimen Preparation

Forty freshly extracted non-endodontically treated human maxillary canines were extracted for periodontal reasons. Inclusion criteria were absence of severe root curve, decay, and cracks; existence of at least a 14-mm root length from cemento-enamel junction to apex. Exclusion criteria were completely oval and wide canals and defect formation in specimens during preparation. All external debris was eliminated with an ultrasonic scaler. Teeth were stored in a 0.9% saline solution at 4°C for no longer than four months. To disinfect, specimens were immersed in 2.5% NaOCl for 15 minutes. Each tooth was marked at a 14-mm distance from the apex. The crown portion of each tooth was removed at this mark with a diamond disk under water cooling. The root canals were then shaped to size 30 with a 0.06 taper (K-Files, Maillefer, Ballaigues, Switzerland) and, after irrigation with normal saline, were filled with gutta-percha (Diadent, Tianjin, China) and a non-eugenollic sealer (AH26, Dentsply-Maillefer). Specimens were fixed vertically and embedded in autopolymerizing clear acrylic resin (Triplex, Ivoclar Vivadent, Schaan, Liechtenstein) surrounded by a wax mold. Before post placement, excess gutta-percha was removed with Gates Glidden burs #3 (Gates Drills, Mani Inc, Utsunomiya, Japan), leaving 4 mm of it intact in the apical portion. Prefabricated conical shape glass fiber posts #3 (FRC Postec Plus, Ivoclar Vivadent) were used in this study. Their polymer matrix was composed of aromatic and aliphatic dimethacrylates. They also contained ytterbium trifluoride. The root canals were then prepared with a reamer #3 that was available in the FRC Postec Plus kit. The prepared spaces were rinsed with 2.5% NaOCl to remove the smear layer from the root canal walls. Final irrigation was done with normal saline; excess water was then removed with air and paper points. Posts were cut horizontally at a mark made 10 mm from the end. After the complete seating of posts was confirmed, they were divided into five groups (n=8) and prepared in different manners. In group HF+S (HF + silane), the posts were exposed to 9.5% HF (Porcelain Etchant, Bisco, Schaumburg, IL, USA) for 60 seconds, irrigated with water for 15 seconds, then gently air-dried. To enhance chemical bonding, a silane solution (Bis-silane, Bisco) was applied on the post surface in two layers by a microbrush (Multi-brush Multi-colors, Denbur Inc, Oak Brook, IL, USA) and gently air-dried for 60 seconds at room temperature following the manufacturer's instructions. In group HF+S+WP (HF + silane + warmed post), pretreatment of posts was performed as explained for group HF+S followed by immersion of the silanated posts in warm water (45°C) for 10 seconds and then gentle air-drying with warm air. The water temperature was adjusted and fixed with a Hanau Compound Heater (Hanau Engineering Co, Inc, Buffalo, NY, USA). Warm air (45±5°C) was generated from a blow dryer (Heat-Blo Guns, Fisher Scientific, Pittsburgh, PA, USA) blown onto the silane applied to the post surface for 30 seconds. In group H_2O_2+S (H_2O_2 + silane), the posts were immersed in 10% hydrogen peroxide for 20 minutes, followed by the same procedures as described for group HF+S. In group H_2O_2+S+WP (H_2O_2 + silane + warmed post), pretreatment of the posts was the same as for group H_2O_2+S. The final procedure was immersion of silanated posts in warm water (45°C) for 10 seconds and then gentle air-drying with warm air. In group C, no post surface pretreatment was carried out.

A self-etch resin cement (Panavia F2.0, Kuraray, Osaka, Japan) was used to cement the posts. Equal amounts of ED Primer II liquids A and B (Kuraray) were mixed and applied to the prepared root space with a microbrush tip for 30 seconds; the excess was removed by paper points. Equal amounts of Panavia F2.0 base and catalyst paste were mixed on a pad with a plastic instrument and applied to the prepared space with a lentulo spiral #30 (Maillefer). The posts were then coated with luting cement and inserted into the prepared canal spaces using finger pressure. The excess cement was immediately removed with a microbrush. The luting agent was polymerized with a light curing unit (Demi-LED Light curing system, Kerr Corp, Orange, CA, USA) with 400 mW/cm^2 output for 40 seconds. The specimens were stored in water at room temperature for 24 hours and subjected to thermal cycling (1000 cycles, 5°-55°C, 30-second dwell time).

Push-out Testing

Each specimen was sectioned perpendicular to its long axis with a slow speed diamond saw (Accutom-50, Struers, Copenhagen, Denmark). After discarding the first 0.5-mm slice, three 2-mm-thick sections of each root were obtained. The thickness of each slice was measured and recorded using a digital caliper (SC-6 Digital Caliper, Mitutoyo Corp, Tokyo, Japan) with an accuracy of 0.01 mm. Each specimen was positioned and fixed onto the stainless steel jig, which had a central hole to support the dentin and acrylic resin. A compressive load was applied apico-coronally on the apical surface of each slice by a cylindrical metallic pin on a Universal Testing Machine (Zwick Roell Z020, Zwick, Germany). Because of the tapered type of the post, three different pin sizes (1.1, 0.9, and 0.7 for the coronal, middle, and apical sections, respectively) were used for push-out testing. The load was applied with a crosshead speed of 0.5 mm/min until failure occurred. Push-out bond strength was expressed in megapascals (MPa) by dividing the load at failure

Table 1: *Mean Push-out Bond Strengths (MPa) and Standard Deviations (SDs) for Experimental and Control Groups*[a]

Group[a]	Mean (SD)			
	Root Levels			Total[b]
	Apical	Middle	Coronal	
HF+S	6.94 (4.07)	8.43 (1.65)	9.34 (3.58)	8.24 (1.49)[y]
HF+S+WP	14.64 (4.57)	10.96 (7.9)	12.08 (2.98)	12.56 (1.73)[z]
H_2O_2+S	6.44 (2.11)	6.41 (4.68)	11.64 (2.62)	8.16 (1.38)[y]
H_2O_2+S+WP	6.76 (1.89)	9.69 (2.59)	14.38 (10.51)	10.28 (3.57)[yz]
Control	9.13 (2.55)	10.53 (2.23)	6.96 (5.52)	8.87 (1.31)[y]

[a] *HF+S, hydrofluoric acid + silane; HF+S+WP, hydrofluoric acid + silane + warmed post; H_2O_2+S, hydrogen peroxide + silane; H_2O_2+S+WP, hydrogen peroxide + silane + warmed post; control, no pretreatment.*
[b] *Means with different superscript letter show a statistically significant difference between groups (p<0.05).*

(Newtons) by the surface area (mm^2). The total bonding area for each segment was calculated using the following formula:

$$\pi(R + r)[h^2 + (R - r)^2]^{1/2}$$

(R=Radius of the canal at the coronal surface of the slice, r=radius of the canal at the apical surface of the slice, h=height of the slice.)

Failure Mode Evaluation

After the posts were dislodged, they were observed under a stereomicroscope (Lomo SF-100, MBC-10, Moscow, Russia) (36×), to determine the type of failure. Failures were classified in three groups: 1) cohesive failure in post or dentin, 2) adhesive failure between the resin and post or the resin and dentin, and 3) mixed failure.

Scanning Electron Microscope Analysis

Four posts were selected in order to evaluate their surface conditions after acid etching. For this evaluation two posts were immersed in 10% H_2O_2 for 20 minutes, and two posts were exposed to 9.5% HF for 60 seconds. The prepared specimens were then immersed in 96% ethanol and gently air-dried. Each post was sputter-coated with gold alloy (SCD 005 Sputter coater, Bal-Tec Co., Balzers, Vaduz, Liechtenstein, Germany) and analyzed under a scanning electron microscope (SEM, Philips XL30, Philips Eindhoven, Netherlands).

Statistical Analysis

The two-way analysis of variance (ANOVA) was used to evaluate the effect of independent factors (different chemical etching and heat treatment of silane solution) on the dependent variable (push-out bond strength). Moreover, post-hoc Tukey honestly significant difference (HSD) test and paired sample t-test were used. The significance level was set at α=0.05.

RESULTS

The mean and standard deviation (SD) values of the push-out bond strength for each of the five groups are shown in Table 1. Two-way ANOVA revealed that the type of chemical agent used for etching the post surface had no significant effect on bond strength (p=0.224), whereas the bond strength was significantly increased via heat treatment of the silane solution using warm water ($p<0.001$). The interaction between these two factors was not statistically significant (p=0.142).

Use of HF for etching in combination with the heat treatment of the silane solution produced the best overall results. The Tukey HSD test displayed significant differences between group HF+S+WP and group HF+S (p=0.002) and between group H_2O_2+S (p=0.001)and group C (p=0.009). The difference between group HF+S+WP and group H_2O_2+S+WP was not significant (p=0.20). By use of the paired sample t-test between the four pretreated groups and ignoring the control group, the coronal third of the root (10.88±6.08 MPa) showed significantly greater bond strength than the middle third (9.21 ± 4.53 MPa) and the apical third (8.78 ± 4.36 MPa) at p=0.05 and p=0.022, respectively.

Evaluation of the failure mode revealed that the most frequent failure was mixed (77.5%), followed by adhesive failure (14%), and cohesive failure in the post (8.5%). There was no cohesive failure in dentin.

SEM Analysis

SEM evaluation revealed that the post surface morphology was altered after etching with 9.5% HF and 10% H_2O_2. The surface treatments dissolved the post resin matrix and created microspaces among

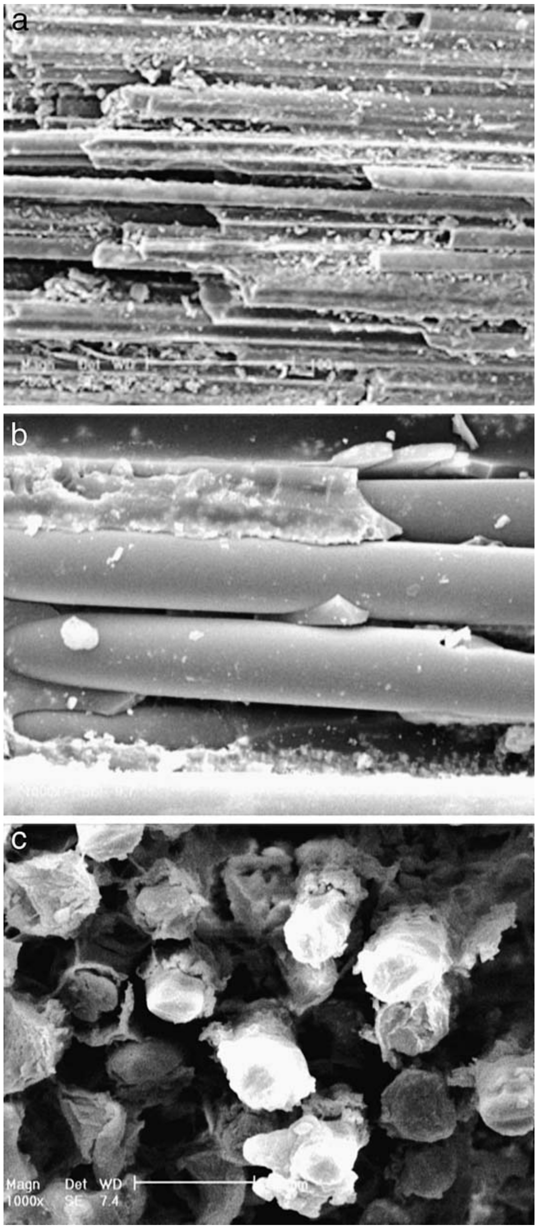

Figure 1. *SEM photomicrographs of the post surface and cross-section of the post treated with 9.5% HF. (a): Post surface, 200×, bar = 100μm. (b): Post surface 1000×, bar = l0μm. (c): Cross-section of the post, 1000×, bar = l0μm.*

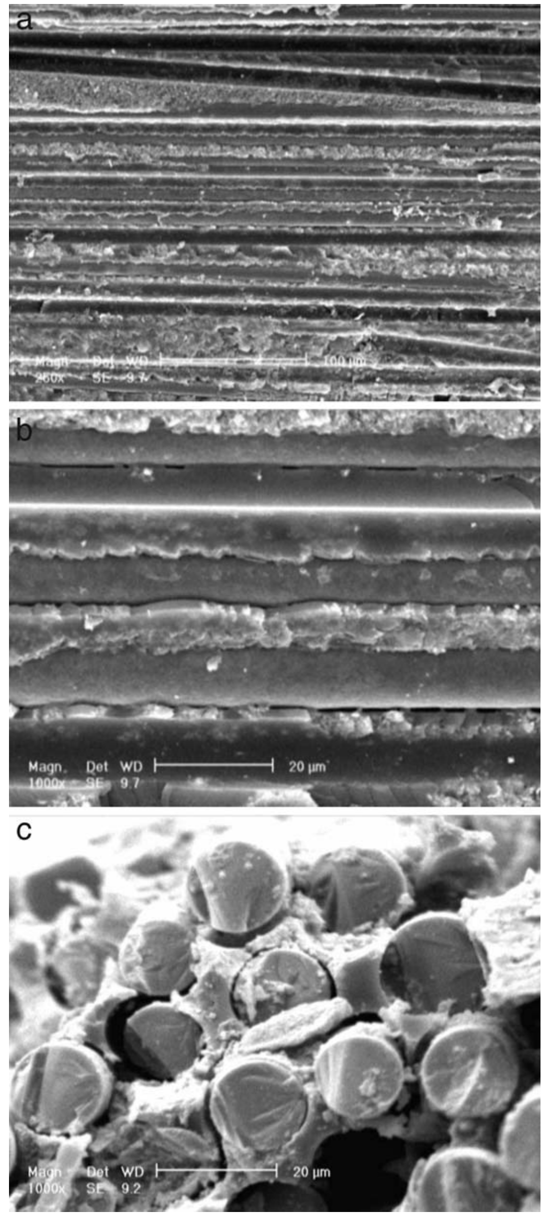

Figure 2. *SEM photomicrographs of the post surface and cross-section of the post treated with 10% H_2O_2. (a):.Post surface, 200×, bar = 100μm. (b): Post surface 1000×, bar = l0μm. (c): Cross-section of the post 1000×, bar = l0μm.*

the exposed fibers. Treatment with 9.5% HF had a greater impact on the post surface. In the cross-section view it was revealed that more superficial fibers were exposed with the HF pretreatment because larger amounts of the resin matrix were removed to a greater depth (Figures 1 and 2).

DISCUSSION

The present study evaluated the push-out bond strength of a glass fiber post to the root canal dentin with different surface pretreatments. There were no significant differences between the groups prepared with HF and those prepared with H_2O_2. The second null hypothesis was accepted. Based on the SEM analysis, treatment with 9.5% HF dissolved the resin matrix more extensively and to a greater depth than did treatment with 10% H_2O_2. Because of the more corrosive nature of HF, dissolution of resin matrix and glass fibers creates a rough surface that causes penetration of resin composite into the microporosities and leads to an increase in bond strength. However, the etching effect of H_2O_2 depends on its capacity to partially dissolve the resin matrix, breaking epoxy resin bonds and exposing the surface of fibers to silanization through a mechanism of substrate oxidation.[15,19] This effect, in which only the epoxy resin is dissolved with no effect on glass fibers, may reflect the insignificantly lower bond strength of H_2O_2 groups. These findings are in contrast to the results of the study by Vano and others,[19] which reported an increased bond strength after H_2O_2 application compared with HF application. Several reasons, such as type of test performed, could be responsible for these conflicting results. Vano and colleagues used microtensile testing to evaluate the bond strength of different composite core buildups and pretreated fiber posts. In our study we used push-out testing, which has been shown to provide a better estimation of bond strength in the root canal than conventional shear tests do.[1] Moreover, the large and complex C-factor (ratio of bonded to nonbonded surfaces) in long, narrow post spaces is a complicating factor resulting in formation of numerous interfacial gaps.[6] Several factors may be associated with the detrimental effect of H_2O_2 on bond strength; H_2O_2 is capable of generating the hydroxyl radical, an oxygen-derived free radical, and leaves behind an oxygen-rich surface that inhibits polymerization of resin.[20,21] The formation of such a surface in the narrow space of the root canal and nonreleased stress may have led to the lower bond strength in the root region in the present study.

Voids and air bubbles may prevent proper cementation of the post, thus causing its debonding. D'Arcangelo and others[22] reported that using the Lentulo spiral to apply the luting resin enhances bond strength. In the present study, the resin cement was inserted in the canal with a Lentulo spiral and applied on the post surface simultaneously to reduce voids and increase the displacement resistance of fiber posts.

Thermocycling has been considered an essential aspect of dentin adhesion testing. It has been shown that thermocycling results in a significant decrease in the flexural strength of fiber posts.[23] Purton and others[4] reported no significant differences between thermocycled and non-thermocycled specimens in regards to the forces required to cause post-retention failure. Nevertheless, in the present study thermocycling was used to simulate the clinical condition.

Silanes improve the bond of composite resin to porcelain; however, the use of silanes to enhance bond strength between composite resin and the fiber post is controversial.[23] Silanes provide the chemical bond between the inorganic matrix of luting agent and the organic surface of the fiber post, protect the fibers from damage during handling, and improve the catalytic and wettability characteristics of the fiber post surface. Moreover, they enhance the chemical resistance of the fiber resin interface to hydrolysis.[18] The results of this study showed that silane heat treatment significantly increases bond strength ($p<0.001$). Thus, the first null hypothesis can be rejected. Similar results have been reported by Monticelli and others.[15] In their research, the bond strengths of a two-component silane and a one-component ethanol-based silane were increased by heat treatment via application of warm air. For optimal adhesion to the fiber post structure, only a monolayer of the silane is required to convert the ceramic surface from an Si-OH to a methacrylate appearance, but usually multiple layers form.[25] A thicker silane interphase may become the weak link of the bond. The innermost layer is cross-linked and provides a strong siloxane bond, whereas the outermost and intermediate layers are only physically adsorbed to the innermost layer and contain many oligomers, which can be easily washed away by organic solvents or water.[26] To provide heat treatment and removal of the weakly bound oligomers, in this study warm water was used to wash the post surface after silane treatment. This technique may produce a bond that is much more hydrolytically stable than if the silane were simply applied and left to dry.

Because of the dependence of core stability to post-root adhesion, the bond strength evaluation at each level of the root seems to be important; therefore, one of the aims of the present study was to compare the bond strength at different levels of the root. It seems that the post retention in the root canal greatly depends on frictional sliding rather than micromechanical and chemical adhesion.[6] In this study, the conical FRC Postec Plus posts were used to eliminate the effect of sliding friction. Although a special reamer with a similar-sized fiber post was used, complete adaptation in the coronal third was impossible. In areas where there is good adaptability between the post and dentin, sliding friction and micromechanical interlocking are important; in areas where the adaptability is low, chemical bonding could be more effective.

This study was performed with the purpose of improving the bond strength between the post and cement by use of different post surface treatments.

In the present study mixed failure was the most frequent type of failure, followed by adhesive failure, between the post and cement. This finding is consistent with the result reported by D'Arcangelo and others,[22] which confirmed that the post-cement interface is weaker than the dentin-cement interface, and special attention should be given to it.

CONCLUSION

Based on the results of this *in vitro* study, heat treatment of silane had a significant effect on the push-out bond strength. There were no significant differences in the mean bond strength between pretreatment with HF and pretreatment with H_2O_2.

Acknowledgements

The study was approved by the Medical Ethics and Research Office at the Isfahan University of Medical Sciences and financially supported by this university.

Note

This report is based on a thesis that was submitted by Fahime Salamt to the School of Dentistry, Isfahan University of Medical Sciences, Isfahan, Iran, in partial fulfillment of the requirements for the MSc degree in operative dentistry.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 8 October 2013)

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