Original Article

Effect of Silane Solvent on Microtensile Bond Strength of Hydrogen Peroxide-Treated Fiber Post and Composite Core

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Abstract:

Objective: The aim of this *in vitro* study was to evaluate the effect of the type of solvent in silane solution on microtensile bond strength of fiber posts to composite resin cores after application of 24% hydrogen peroxide.

Materials and Methods: Eighteen white fiber posts, immersed in 24% hydrogen peroxide were divided into three groups (n=6). In the group A post surfaces were silanized with an ethanol based solution, in group B with an acetone based solution, in the group C with and un-diluted methacryloxytrimethoxysilane (as the control group). The cores were built up using flowable composite. Microtensile bond strength test and evaluations using stereomicroscope were performed on the samples and the data were analyzed using one-way ANOVA and Tukey HSD tests.

Results: A significant difference was observed between the amounts of microtensile bond strength of fiber posts to composite cores in the groups A and B, and the ones in group C ($P<0.05$). There was no such difference between groups A and B ($P>0.05$).

Conclusion: The type of solvent in silane solution has no effect on microtensile bond strength between fiber post and composite resin core after application of 24% Hydrogen Peroxide.

Key Words: Silane, Solvent; Fiber Post; Composite Core, Hydrogen Peroxide, Endodontics; Treated Teeth

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INTRODUCTION

In recent years, clinical use of fiber reinforced composite (FRC) posts has been increased as alternatives to cast metal posts in restoration of endodontically treated teeth [1]. Posts should be indicated for core retention and should be supported the cemented crowns [2].

A variety of resin composite materials is available to the clinicians for core build up [3]. Improvement of adhesion at the interface between the composite core and the fiber post is one of the important factors for durability of the final restoration. Surface treatments are common methods to provide chemical and/or mechanical interaction between the post and the surrounding composite. Available techniques such as phosphoric and hydrofluoric acid etching, sand blasting with alumina particles and using CoJet with silica-coated alumina particles facilitate chemical and micro-

mechanical retention between different substrates.

Treatment with 24% hydrogen peroxide and 21% sodium ethoxide and etching with potassium permanganate solution are also introduced to enhance the surface roughness. It has been reported that etching with hydrogen peroxide provides an easy, effective, and conservative method for surface treatment in comparison with other corrosive liquids [4]. Hydrogen peroxide partially dissolves the surface epoxy resin and breaks its bond through a mechanism of substrate oxidation without damaging the quartz fibers [5-8].

Another method of treatment is applying a silane solution. Silane coupling agents are hybrid organic-inorganic compounds that can mediate adhesion through intrinsic dual reactivity, improve wettability of the quartz fibers, provide strong bond between the fibers and the composite and increase the chemical resistance of the fiber-matrix interface especially to water [9,10].

Monticelli et al [3] showed that surface treatment of the resin phase of fiber posts with hydrogen peroxide enhanced the silanization efficiency of the quartz fiber phase so that the acquired adhesion in the post/core system is the sum of chemical and micro mechanical retention. An *in vitro* study revealed that bond strength values of silanized and hydrogen peroxide-treated fiber posts to resin cores are greater than only silanized ones [11].

It has also been previously depicted that the type of the silane affects the retention of the core to the post surface [12,13], but no data are available on the role of different solvents of silane solutions on the adhesion between the fiber post and the core when the post surface is pre-treated with hydrogen peroxide.

This study evaluates the effect of the solvent in the silane solution on the bond strength between fiber post and composite core after pretreating the post surface with a 24% hydrogen peroxide solution.

MATERIALS AND METHODS

Ethanol (10 wt.% of deionized water+90 wt.% of ethanol, Merck, Germany) and acetone based (10 wt.% of deionized water+90 wt.% of acetone, Merck, Germany) solvents were prepared. pH of the solvents was adjusted to about four by addition of a few droplets of acetic acid (Merck, Germany) and monitored with a pH-meter (Inolab level 2, WTW, Germany). Silane solutions were then prepared incorporating 1 wt.% of γ-methacryloxypropyl trimethoxy silane (γ-MPS) into the ethanol and acetone based solvents.

The solutions were kept for one hour at room temperature to ensure full hydrolysis of the silane. Eighteen white posts (#2, RTD, St. Egèven, France), maximum 1.8 mm in diameter and made of unidirectional pretended quartz fibers and embedded in an epoxy resin matrix were first immersed in a 24% hydrogen peroxide solution for 10 min at room temperature, rinsed under running water for two minutes, gently air-dried and divided into three groups (n=6).

In groups A and B, post surfaces were silanized for 60 seconds using the ethanol and the acetone based silane solutions, respectively. In group C (as the control group), solvent-free silane (γ-MPS) was used for the purpose. Having applied the silane, the posts in the group C were stored for one hour due to probable prehydrolyzation of the silane and the reaction between OH groups in the silane and those on the post surfaces.

Core build up was then performed using flowable composite (Ælite Flo, Bisco, USA). Each post was positioned upright on a glass slab and fixed with a drop of sticky wax. A cylindrical matrix was placed around it and adjusted so that the post would be exactly in the center. The matrix was 10 mm in diameter and the length was equal to the non-tapered portion of the post (6 mm). Flowable composite was applied on the post in one-millimeter thickness increments and cured separately using a halo-

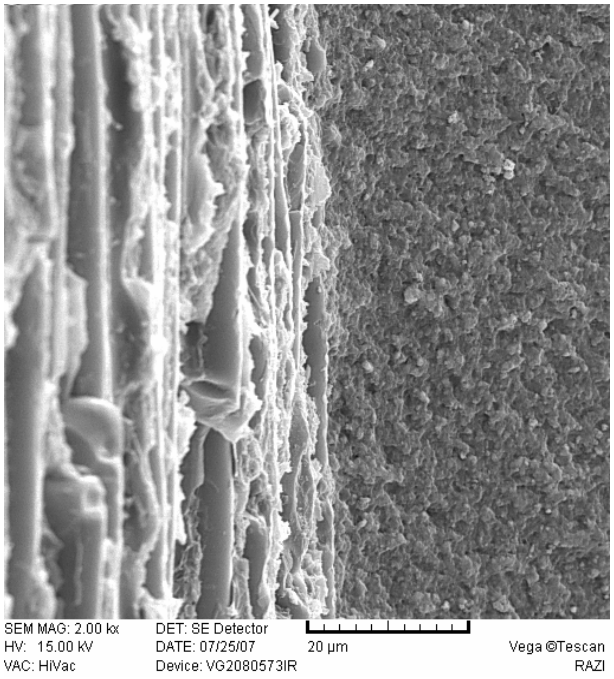

Fig 1. SEM image of the post surface in Group A; (2000x).

gen light curing unit (450 mW/cm^2, Degulux II, Degussa AG, Gschttsbereich Dental, D-63457 hanau-Wolfgang, Germany). To ensure optimal polymerization of the core material an additional 40s curing was performed from the bottom of the cylinder prior to removal of the matrix.

By means of a water-cooled blade of a sectioning machine (Hamco Inc., Rochester, NY), one-millimeter thick sections were serially cut perpendicular to the long axis of the post, and two longitudinal cuts were made. Finally, sticks with uniform thickness were prepared, in which the posts were placed in the center and the core build-ups on each side (12 sticks for each group). Every stick was glued to the two free sliding components of a jig mounted on the micro tensile tester (Bisco, USA) with mad wolf. This set-up was designed to apply pure tensile forces to the two opposite post-core interfaces. The specimens were loaded at a crosshead speed of 0.5 mm/min until failure occurred at either one of the two stressed interfaces. Bond strength was expressed in Mega Pascal (MPa), by dividing the load at failure (Newton) to the bonding surface area (mm^2).

Failure modes of the samples were assessed using a stereomicroscope (M6C-10, Russia) at x7 magnification. The following items were used for evaluation: cohesive failure (failure in post or core material), adhesive failure (failure at the interface of post and core material), and mixed failure (adhesive-cohesive failure).

Scanning electron microscopy (SEM, Tescan, ZEGA II, SMU, Czeck Republic) evaluation of the fractured surfaces was performed on two randomly selected fractured sticks of each group. The data were analyzed using one-way ANOVA and Tukey HSD tests at 5% level.

Table 1. Microtensile bond strength of post and core.

Groups	N	Mean (SD)
A (Ethanol-based silane solution)	12	13.16 (4.83)
B (Acetone-based silane solution)	12	12.13 (3.88)
C (Solvent-free silane solution)	12	7.04 (3.05)

RESULTS

Means and standard deviations of the microtensile bond strength (MTBS) of groups A, B and C were 13.16 (SD=4.83), 12.13 (SD=3.88) and 7.04 (SD=3.05) MPa, respectively. One-way ANOVA test indicated a significant difference among the MTBS amounts of the three groups. Paired comparisons by Tukey test showed a significant difference between the amounts of group C, in which the posts were silanized with the pure solvent-free γ-MPS,

Table 2. Paired comparisons of the microtensile bond strength of the tested groups.

Group 1	Gruop2	Mean (SE)	P-Value
C (Solvent-free silane solution)	**A (Ethanol-based silane solution)**	-5.76 (1.73)	0.006
C (Solvent-free silane solution)	**B (Acetone-based silane solution)**	-4.74 (1.80)	0.035
A (Ethanol-based silane solution)	**B (Acetone-based silane solution)**	-1.03 (1.73)	0.820

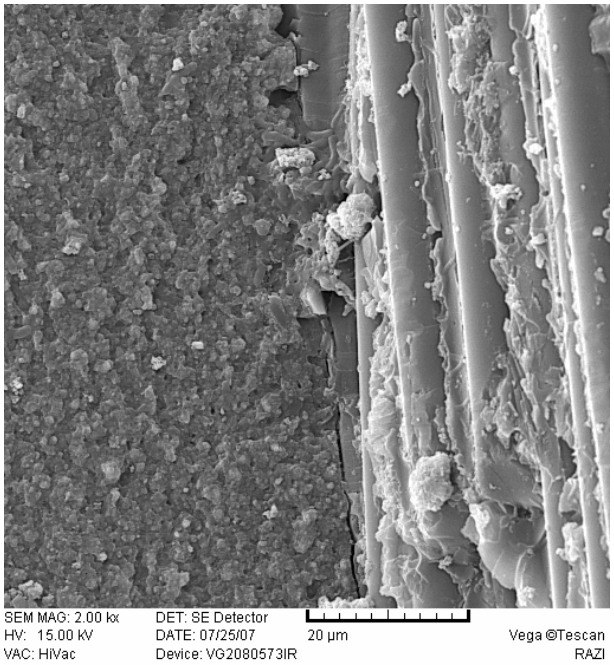

Fig 2. SEM image of the post surface in Group B; (2000x).

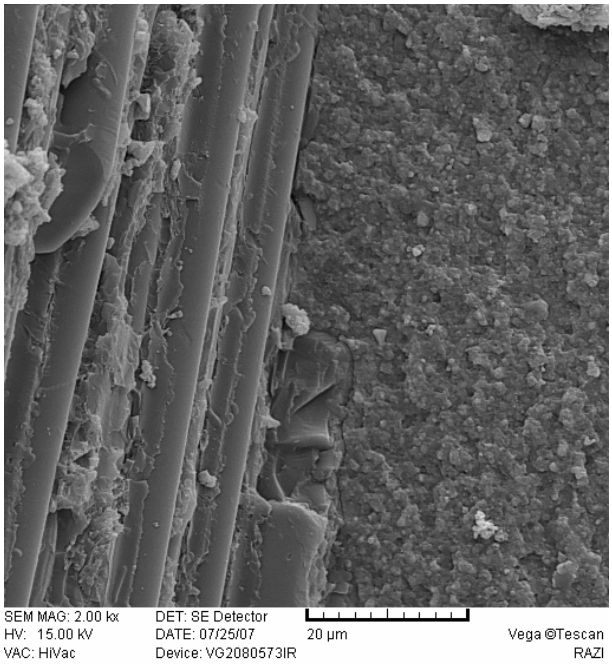

Fig 3. SEM image of the post surface in Group C; (2000x).

and group A and B ($P>0.05$) but there was no such difference between the amounts of groups A and B ($P<0.05$) which were treated with the solvent-based silanes (Table 1).

Stereomicroscope evaluation revealed that all of the failures occurred at the interface of post and core material (adhesive failure).

SEM analysis confirmed the stereomicroscope observations and also showed that the quartz fibers were exposed after treatment with hydrogen peroxide (Fig 1-3).

DISCUSSION

This study evaluated the effect of the type of the solvent in silane solutions on MTBS between fiber posts and composite cores and the results exhibited that MTBS of the specimens treated with solvent-based silane solutions were higher than those treated with the solvent-free silane.

Nowadays, the use of prefabricated FRC posts is popular due to their bond to tooth tissues, low risk of root fracture, and the ability to be removed by special drills. As long-term clinical services of the final restoration depends on adhesion between the posts and composite cores, increasing bond strength of posts to cores contributes greatly to a successful restoration [14,15].

Application of hydrogen peroxide on the surfaces of FRC posts is a reliable, conservative, and easy technique for improving the bond strength. In addition, it has been demonstrated that treatment with hydrogen peroxide in combination with silane can results in more effective adhesion [4]. In our study, to maximize the storage stability, it was preferred that the water be acidified with a water-soluble acid before it was combined with the remaining ingredients of the composition. A preferred pH range for the water is believed to be about two to six, more preferably about three to five for the purpose [16]; therefore, Acetic acid was used to control the pH of the solution adjusted at four.

The lower MTBS amounts of solvent-free silane treated specimens can be attributed to deficient wetting of post surfaces due to lack of

solvent in the solution. Solvent-free silane is more viscous than silane solutions, thus, forming a thick layer of silane on the substrate. It has been shown that the formation of a multilayer structure may result in a reduction of the effectiveness of silane coupling agents and the occurrence of cohesive failure within the silane structure [11,17].

Furthermore, polar solvents help silane solutions to wet high-energy post surfaces and provide a thin layer of silane coated on the posts.

Silane, must ideally be fully hydrolyzed so that all of its hydrolyzable alkoxy groups are changed to silanol groups. If silane is incompletely hydrolyzed, after being coated upon a substrate and dried, it will not bond to the substrate. On the other hand, increasing the degree of hydrolysis in the solution also increases the tendency of the silanol groups to undergo self-condensation. Commercial silane solutions are often not completely hydrolyzed. In two part products, a 15 min standing time after mixing is adequate to ensure full hydrolysis at room temperature. For one-part products, the degree of hydrolysis depends on technique, ambient humidity and the amount of moisture and acid present on the substrate. This results in a poorly controlled and possibly incomplete degree of hydrolysis [16]. Lack of suitable conditions for hydrolyzation of the solvent-free silane is another reason that may explain the lower bond strength values in this group.

The results also showed that there was no significant difference between bond strength values of ethanol and acetone-based solutions.

Alkoxy groups in a silane are hydrolyzed in the presence of water, which can be either in the solution or on the surface of the substrate. Non-aqueous solvents facilitate dissolving of non-hydrolyzed silanes in the solution as well as improving the wetting of the post surface. It seems that both ethanol and acetone-based solvents provide this purpose. It is also evident that 10 wt.% of water in the solution is enough for pre-hydrolyzation of the silanes. Therefore, no difference was observed between bond strength values of the polar protic alcohol-based and the polar aprotic acetone-base silane solutions when hydrogen peroxide was used for post surface etching.

Due to lack of enough functional groups and the low surface energy, bonding of γ-MPS to the epoxy matrix is not good; consequently, it does not provide sufficient bond strength between the epoxy matrix of the post and the methacrylate-based resin of the composite core. With the removal of the superficial layer of epoxy resin via chemical treatment, more exposed quartz fibers, in terms of surface area, are available for reacting with silane molecules [3]. The increased chemical union between silanized quartz fibers and the methacrylate-based core material would significantly improve the interfacial bond strength [4].

Although dental bonding agents are sometimes used in combination with silane coupling agents, Vano et al [11] showed that the adjunction use of an adhesive only produced limited improvement in coupling of resin composites and resin-based fiber posts. Silane coupling agents mainly function by bonding chemically to the post and the core material improving surface wettability. Stereomicroscope and SEM evaluations showed that all the failures were at the post-core material interface, so, the weakest bond strength was at the adhesion area of the two materials.

CONCLUSION

The type of the solvent in the silane solution did not affect the bond strength; however, MTBS of the specimens treated with solvent-based silane solutions were higher than those treated with the solvent-free silane.

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REFERENCES

1-Goracci C, Raffaelli O, Monticelli F, Balleri B, Bertelli E, Ferrari M. The adhesion between prefabricated FRC posts and composite resin cores: microtensile bond strength with and without post-silanization. Dent Mater 2005 May;21(5):437-44.

2-Perdigão J, Gomes G, Lee IK. The effect of silane on the bond strengths of fiber posts. Dent Mater 2006 Aug;22(8):752-8.

3-Monticelli F, Toledano M, Tay FR, Cury AH, Goracci C, Ferrari M. Post-surface conditioning improves interfacial adhesion in post/core restorations. Dent Mater 2006 Jul;22(7):602-9.

4-Monticelli F, Toledano M, Tay FR, Sadek FT, Goracci C, Ferrari M. A simple etching technique for improving the retention of fiber posts to resin composites. J Endod 2006 Jan;32(1):44-7.

5-Holm R, Farrants GW, Nesland JM, Sobrinho-Simões M, Jørgensen OG, Johannessen JV. Ultrastructural and electron immunohistochemical features of medullary thyroid carcinoma. Virchows Arch A Pathol Anat Histopathol 1989;414(5):375-84.

6-Eneström S, Kniola B. Resin embedding for quantitative immunoelectron microscopy. A comparative computerized image analysis. Biotech Histochem 1995 May;70(3):135-46.

7-Brorson SH, Hansen AR, Nielsen HZ, Woxen IK. A comparative study of the immunogold labeling on H(2)O(2)-treated and heated epoxy sections. Micron 2001 Feb;32(2):147-51.

8-Baskin DG, Erlandsen SL, Parsons JA. Influence of hydrogen peroxide or alcoholic sodium hydroxide on the immunocytochemical detection of growth hormone and prolactin after osmium fixation. J Histochem Cytochem 1979 Sep;27(9):1290-2.

9-Matinlinna JP, Lassila LV, Ozcan M, Yli-Urpo A, Vallittu PK. An introduction to silanes and their clinical applications in dentistry. Int J Prosthodont 2004 Mar-Apr;17(2):155-64.

10-Pegoretti A, Fambri L, Zappini G, Bianchetti M. Finite element analysis of a glass fibre reinforced composite endodontic post. Biomaterials 2002 Jul;23(13):2667-82.

11-Vano M, Goracci C, Monticelli F, Tognini F, Gabriele M, Tay FR, et al. The adhesion between fibre posts and composite resin cores: the evaluation of microtensile bond strength following various surface chemical treatments to posts. Int Endod J 2006 Jan;39(1):31-9.

12-Matinlinna JP, Ozcan M, Lassila LV, Vallittu PK. The effect of a 3-methacryloxypropyltrimethoxysilane and vinyltriisopropoxysilane blend and tris(3-trimethoxysilylpropyl)isocyanurate on the shear bond strength of composite resin to titanium metal. Dent Mater 2004 Nov;20(9):804-13.

13-Valandro LF, Filho OD, Valera MC, de Araujo MA. The effect of adhesive systems on the pullout strength of a fiberglass-reinforced composite post system in bovine teeth. J Adhes Dent 2005 Winter;7(4):331-6.

14-Aksornmuang J, Foxton RM, Nakajima M, Tagami J. Microtensile bond strength of a dual-cure resin core material to glass and quartz fibre posts. J Dent 2004 Aug;32(6):443-50.

15-Park SJ, Jin JS. Effect of silane coupling agent on interphase and performance of glass fibers/ unsaturated polyester composites. J Coll Inter Sci 2001 October;242(1):174-9.

16-Culler SR, inventor; Stable silanol priming solution for use in dentistry. United States patent 4673354. 1987 June 16.

17-Debnath S, Wunder SL, McCool JI, Baran GR. Silane treatment effects on glass/resin interfacial shear strengths. Dent Mater 2003 Jul;19(5):441-8.