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In Vitro Hydrolytic Degradation of Composite Quartz Fiber-post Bonds Created by Hydrophilic Silane Couplings

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Clinical Relevance

Silane-based composite bonds are susceptible to hydrolytic degradation: interfacial water sorption induced by hydrolytic couplings may adversely affect their strength.

SUMMARY

The hydrolytic stability of a pre-activated MPS silane (Monobond-S) and a 2-component 4-META/γ-MPTS silane (Porcelain Liner M) to H_2O_2-etched quartz fiber posts was investigated using a modeling approach. Composite build-ups around silanized posts were stored dry for 24 hours, stored in deionized water at 37°C for 24 hours, 1 week, 1 month or they were thermocycled. Sectioned specimens were prepared for microtensile bond testing and SEM examination; 4-META/γ-MPTS silane produced a rapid decline in bond strength after 1 week and 1 month of water storage and after thermocycling. This was not apparent in pre-activated MPS silane. SEM revealed debonding along the post-composite interfaces, which were coupled with 2-component silane. The use of a hydrophilic resin monomer (4-META) for on-demand hydrolysis of the γ-MPTS silane expedited interfacial water sorption and hydrolytic degradation, which may be prevented with alternative coupling strategies.

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INTRODUCTION

Concern over the integrity of the apical seal after preparation of post spaces in endodontically treated teeth has stimulated research that compared the effects of immediate versus delayed post space preparations on apical leakage.[1-2] However, little has been reported in the endodontic literature on the hydrolytic stability of silane-coupled resin composite-fiber post bonds that contribute to the longevity of the coronal seal in post-supported endodontically treated teeth.

Bifunctional trialkoxy silanes [R'Si(RO)^3] are widely used in fiber-reinforced composite technology.[3] The alkoxy functional groups (RO) of these silane molecules enable them to be anchored to inorganic substrate-bearing hydroxyl groups, while the alkyl functional group (R') improves their compatibility with organic substrates. Copolymerization may also occur between the R' group and organic matrices.[4] Although the use of silane coupling generally improves the wetting of these bonded substrates,[5-6] silanes containing hydrophilic functional groups exhibit a higher susceptibility to water sorption and, over time, undergo a variable extent of hydrolysis.[7-8]

Table 1: *Chemical Composition and Application Mode of the Tested Materials*

Material	Composition	Application
Rebilda DC (Voco, Cuxhaven, Germany)	Bis-GMA; UDMA; BHT; Benzoyl peroxide; organic filler.	Apply in a single bulk. Light cure for 40 seconds.
Monobond-S (Ivoclar-Vivadent, Schaan, Liechtenstein)	3-MPS (1%); Ethanol/water-based solvent; acetic acid.	Apply in a single layer with a brush. Gently air dry after 60 seconds.
Porcelain Liner M (Sun Medical Co Ltd, Moryama, Japan)	Liquid A MMA; 4-META (10%) Liquid B MMA; seconds. γ-MPTS (10%)	Mix Liquid A and Liquid B (1:1). Apply with a brush. Gently air dry after 5 seconds.

BHT: Butylated Hydroxytoluene; UDMA: Urethan dimethacylate monomer; Bis-GMA: Bis-phenol A diglydidylmethacrylate; 3-MPS: 3-methacryloxypropyltrimethoxysilane; MMA: Metyl methacrylate; 4-META: 4-methacryolxyethyl trimellitate anhydride; γ-MPTS: Trimethoxysilyil propyl methacrylate.

MPS silane (3-methacryloxypropyltrimethoxysilane) is commonly used in dentistry due to the compatibility of its methacrylate group with the dimethacrylates components of resin composites.[9] The application of silane coupling agents to quartz fiber posts improves their bond strengths to composite core restorations,[10-11] particularly when the post surfaces are etched prior to silane coupling, creating a more chemically reactive, micromechanically retentive surface.[12-13]

With the growing popularity of direct post and core restorations after root canal treatment, there is a need to evaluate the durability of silane coupling in a wet environment. The thermocycling and water storage of bonded specimens have been widely used for *in vitro* testing of resin-based dental materials to assess the resistance of bonded interfaces to hydrolytic degradation.[14-15] Thus, the objectives of this study were to evaluate, with a model of accelerated water aging, the effects of water storage and thermocycling on the microtensile bond strength of resin composite cores to silanized fiber posts and examine the micromorphologic characteristics of aged interfaces. The null hypothesis tested was that there are no differences in adhesive effectiveness and degradation resistance of 2 different silane coupling agents on fiber post/core build-ups after water storage.

METHODS AND MATERIALS

Seventy quartz fiber posts (DT Light Post Size #2; RTD, St Egéve, France) were etched with 10 vol% hydrogen peroxide (Panreac Quimica, Barcelona, Spain) solution for 20 minutes,[12] sonicated for 10 minutes in deionized water (P Selecta SA, Abrera, Spain), immersed in 96% ethanol (Panreac Quimica) and dried with oil-free air. These quartz fiber posts are composed of unidirectional pre-tensed quartz fibers (60 vol%) embedded in an epoxy resin matrix (40 vol%). They were divided into 2 groups (n=35) according to the silane coupling system applied. The first group consisted of a pre-activated silane (3-methacryloxypropyltrimethoxysilane; 3-MPS) dissolved in an ethanol/water solvent (Monobond-S, Ivoclar-Vivadent, Schaan, Liechtenstein).[10-16] The second group consisted of a 2-component silane system that includes a silane, trimethoxysilyl propyl methacrylate (γ-MPTS) and hydrophilic adhesive resin monomer 4-methacryolxyethyl trimellitate anhydride (4-META) (Porcelain Liner M, Sun Medical Co Ltd, Moryama, Japan)[17-18] which are segregated into 2 separate bottles (Table 1).

Resin composite core build-up was performed with a dual-cured core composite (Rebilda DC; Voco, Cuxhaven, Germany) using a previously reported technique[10] (Table 1). Only the portion of the fiber post with a constant diameter (1.8 mm) was used for the core build-up. Cylindrical specimens were fabricated with the silanized posts in the center of the composite core build-ups. Each experimental group was then divided into 5 subgroups (n= 7): 1) Storage in deionized water at 37°C for 24 hours; 2) for 1 week; 3) for 1 month, with the deionized water changed daily until testing, 4) Thermocycling (Omron Electronics, Madrid, Spain) at 5°/55°C in deionized water for 5000 cycles, with a dwelling time of 30 seconds in each water bath and 5) Dry storage in a desiccator at room temperature for 24 hours (control).

Microtensile Bond Strength Evaluation

Each specimen was mounted in a slow speed diamond saw (Isomet 4000, Buehler, Lake Bluff, IL, USA) and sectioned under water cooling to obtain a slab of uniform thickness, with the post in the center and the core build-up on either side. The slab was further sectioned perpendicular to the original sectioning plane to produce 5-6 beams 1-mm thick. Each beam was glued to

the flat grip of a Bencor Multi-T testing device (Danville Engineering, San Ramon, CA, USA) with cyanoacrylate (Zapit, Dental Ventures of America, Corona, CA, USA) and loaded in tension in a universal testing machine (Model 4411, Instron, Canton, MA, USA) at a crosshead speed of 0.5 mm/minute until failure. Failure modes were classified as adhesive (at the post/composite interface), cohesive (within the post or composite) or mixed (a combination of the 2 modes of failure along the same interface). Interfacial bond strengths were derived using a mathematical formula previously described by Bouillaguet and others[19] for calculation of the curved bonding areas.

The data were analyzed with 2-way ANOVA to evaluate the effects of the aging procedure and silane coupling systems on the microtensile bond strength. Multiple comparisons were performed with the Student-Newman-Keuls test. Statistical significance was set at α=0.05.

Scanning Electron Microscopy (SEM)

Two representative specimens from each silane subgroup were examined with a scanning electron microscope (1430 VP, LEO Electron Microscopy Ltd, Cambridge, UK) to identify the characteristics of the silane coupled interfaces after the different aging procedures. The specimens were sectioned with a water-cooled Isomet saw to obtain 1.5-mm-thick cross sections. The specimens were polished with wet silicon carbide papers, sonicated for 5 minutes in deionized water, rinsed with 96% ethanol, air-dried and sputter-coated with gold. Micrographs were taken using a combination of 50% secondary electron mode (SEI) and 50% back-scattered electron mode (BSE), because of the greater sensitivity of this technique in determining compositional differences between the tested materials.

RESULTS

Microtensile Bond Strength

Both bond strengths and the aging procedure were significantly affected by the silane coupling system (p<0.001). The interaction between these 2 factors was also statistically significant (p<0.012). The 24-hour dry bond strength of Monobond-S was higher than that obtained using the 2-component silane system Porcelain Liner M (Table 2). Most failures were adhesive in nature. However, a small number of cohesive failures within the post were seen in the MPS-treated specimens after a month of water storage.

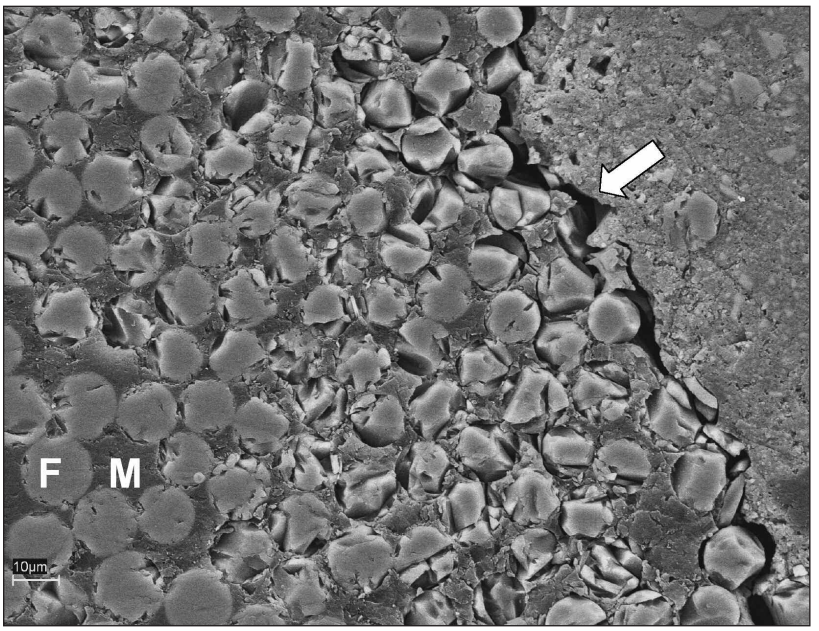

Figure 1. *Representative high magnification SEM micrograph of the post-composite interface after 1 month of aging in deionized water (2500x; bar=10 µm). Lack of bonding between the 4-META/γ-MPTS (Porcelain Liner M) treated post surface and the composite core is evident (arrow) along the entire circumference of the post. C: resin composite; F: quartz fibers within the fiber post. M: Epoxy resin matrix of fiber post.*

Table 2: *Microtensile Bond Strengths of a Core Build Up Composite to Silanized Fiber Posts After Aqueous Aging*

Subgroups	Monobond-S			Porcelain Liner M		
	Bond Strength (MPa)	Failure Mode (%)		Bond Strength (MPa)	Failure Mode (%)	
	Mean (SD)§	Adhesive	Cohesive	Mean (SD) §	Adhesive	Cohesive
24-hour dry	9.43 (2.7) **a**	100%	-	8.88 (2.1) **a**	100%	-
24-hour water	9.44 (3.6) **a**	96%	4% (c)	7.21(2.8) **ab**	95%	5% (c)
1-week water	8.88 (2.3) **a**	100%	-	6.40 (1.8) **b**	100%	-
1-month water	8.92 (2.8) **a**	87%	13% (p)	6.16 (2.2) **b**	100%	-
Thermocycling	8.46 (3.0) **a**	100%	-	6.46 (1.1) **b**	100%	-

(p) = cohesive failure in the post; (c) = cohesive failure in the composite.
§ Subgroups with the same lower case letters are not statistically significant (p>0.05).

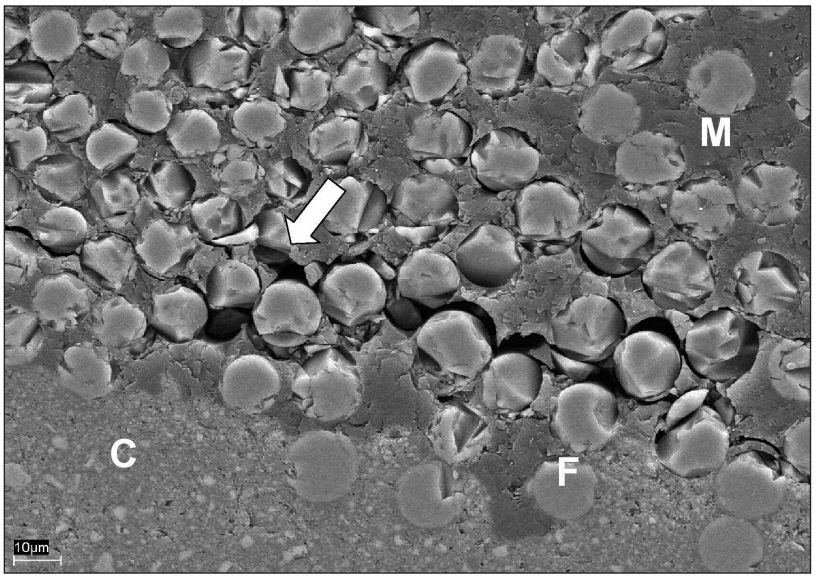

Figure 2. *SEM image of a cross section of a post-composite build-up that was treated with the MPS pre-hydrolyzed silane (Monobond-S) after 1 month of water storage (2500x; bar= 10 µm). Although the interface did not appear to be affected by water sorption, partial delamination of the quartz fibers from the epoxy resin matrix could be seen (pointer). C: resin composite; F: quartz fibers within the fiber post. M: Epoxy resin matrix of fiber post.*

Bond strengths of the Monobond-S subgroups were not affected by any of the aging procedures. Conversely, there were significant declines in bond strength of the 2-component silane Porcelain Liner M after 1 week of water storage and after thermocycling (Table 2). No differences were observed among the 1 week, 1 month and thermocycled subgroups of Porcelain Liner M. The failure mode was predominantly adhesive in nature.

SEM Examination

After water storage, differences in the sites of potential weakness could be identified between the 2 silane groups. For the 2-component 4-META/γ-MPTS silane system, partial debonding occurred between the silane-coupled fiber post and the resin composite (Figure 1). Interfacial debonding was more pronounced with increases in water storage time. Although interfacial debonding did not occur in the MPS specimens, delamination of the quartz fibers from the adjacent epoxy resin matrix could be identified along the periphery of the fiber posts (Figure 2).

DISCUSSION

Acrylic tri-alkoxy silanes are often employed to promote adhesion of methacrylate-containing polymers to silica-containing inorganic substrates.[20] In this study, a pre-hydrolyzed MPS silane solution and a 2-component silane system were examined. In the latter, silane (γ-MPTS) is activated on demand by mixing it with the acidic resin monomer 4-META. As the composite-quartz fiber post bonds created by 4-META/γ-MPTS were more susceptible to degradation after water storage or after thermocycling, the authors had to reject the null hypothesis that there are no differences in adhesive effectiveness and degradation resistance of the 2 silane coupling systems on fiber post/core build-ups after water storage. The results of this work were similar to a previous study that utilized a Micro-shear bond test and accelerated water aging to examine the hydrolytic stability of hydrophilic and hydrophobic silanes on miniature glass fiber rods.[21] In that study, micro-droplets of unfilled dental resins were applied to E-glass fibers treated with MPS, a hydrophilic silane and 10-methacryloxydecyltrimethoxysilane, a hydrophobic silane coupling agent. The authors observed that the interfacial strength of the bonds created by the 2 silanes were similar before accelerated water aging. When the silanized glass fibers containing the bonded resin micro-droplets were exposed to water at 60°C for 24 hours, the interfacial strengths from the hydrophobic silane group remained unchanged, while those from the hydrophilic silane group declined by approximately 50%.[21]

Compared to pre-activated silanes, freshly hydrolyzed silanes are considered more reactive as adhesion promoters.[22] However, the results of this study indicate that bonds created by the freshly hydrolyzed 2-component silane system were more susceptible to degradation after water storage. Although pre-activated MPS silane is considered a comparatively hydrophilic silane with respect to 10-methacryloxydecyltrimethoxysilane,[21] the use of 4-META for on-demand hydrolysis of γ-MPTS silane in the 2-component system renders the latter even more hydrophilic. With 2 carboxyl functional groups,[23] 4-META is a more hydrophilic molecule when compared with the silanes employed. Thus, the inclusion of 4-META in the 2-component silane system could have expedited water sorption and breakdown of the chemical coupling between the glass fibers and the resin composite.[24] When silane is applied to an inorganic substrate, a stable chemisorbed layer and a more volatile physisorbed layer are usually formed. Chemisorption tends to improve with time until the layer becomes hydrolytically stable.[25-26] Enhanced water sorption by incorporating 4-META may hydrolyze the silanol (Si-OH) bonds and promote capillary water flow through the post/composite interface.[22,27] Although interfacial water sorption is usually considered a reversible phenomenon,[28] it eventually becomes irreversible due to progressive separation of the bonded surfaces.[27]

Although pre-activated MPS produced relatively hydrolytically stable composite-fiber post bonds, direct

exposure of the specimens to water for up to 1 month also resulted in partial delamination of the quartz fibers from the epoxy resin matrix along the periphery of the fiber posts.[22,29] Although quartz fibers are comparatively inert to water sorption, these microcracks could have been induced by hydrolysis of the silane employed during the manufacturing of these fiber posts and/or swelling of the epoxy resin matrix after water sorption.[30-31] The term "epoxy resin" is a general descriptor that includes a variety of different monomers with a variable extent of hydrophilic groups that contribute differentially to water sorption and diffusion through polymerized resin matrices.[32-34] Unfortunately, the types of silane and epoxy resin utilized in fiber posts are regarded as proprietary information by the manufacturer and were not available to the authors. The phenomenon may be prevented with the application of more hydrophobic coatings[36] or by selecting alternative strategies of post superficial treatment, such as sandblastings,[36] allowing a more hydrolitically stable micromechanical bonding mechanism. The use of a modeling approach by direct specimen immersion in water represents an accelerated form of water aging.[21] However, there is no long-erm clinical evidence of degradation of post-core build-ups in the pre-prosthetic rehabilitation of ronal seal over a follow-up period of 7 to 11 years.[37] The choice of hydrophilic silane coupling systems to create fiber post-composite bonds should be viewed with reservation in light of the ephemeral hydrolytic stability of these bonds. From a manufacturing perspective, the possibility of performing industrial hydrophobic coatings should be considered to fabricate fiber posts. This would simplify the clinical procedures, avoiding technique-sensitive steps.[38] Future studies should also examine the possibility of using more hydrophobic silanes, such as 10-methacryloxydecyltrimethoxysilane,[21] in the coupling of fiber posts to resin composite cements and whether they could improve the hydrolytic stability of these bonds.

CONCLUSIONS

Based on the results of this study, the following conclusions can be drawn:

1. Hydrophilic silanes are prone to water sorption and hydrolytic degradation. Water storage time has a detrimental effect on silanized fiber post-composite bonds.
2. Interfacial debonding is more pronounced when a 2-component 4-META/γ-MPTS silane system is applied; a delamination of the quartz fibers from the epoxy resin matrix occurs at the periphery of MPS-treated posts.

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