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Effect of temperature on the silane coupling agents when bonding core resin to quartz fiber posts

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ARTICLE INFO

Article history:
Received 21 June 2005
Received in revised form
1 September 2005
Accepted 4 November 2005

Keywords:
Fiber posts
Core build-up
Silane coupling
Air-drying
Solvent evaporation

ABSTRACT

Objectives. To evaluate the effect of different silane agents and air-drying temperatures on bond strength of translucent quartz fibre posts to composite resin.
Methods. The post surface was etched with 10 vol% hydrogen peroxide for 20 min. A two-liquid coupling agent containing 4-methacryolxyethyl trimellitate anhydride (4-META) and γ-trimethoxysilyil propyl methacrylate (γ-MPTS) and two pre-hydrolyzed single component silanes containing 3-methacryloxypropyltrimethoxysilane (3-MPS) and glycid-oxi-propyl-trimetil-oxi-silane (GPS), respectively, were used for treating the fiber posts. Two different post-silanization drying temperatures were applied (21 and 38 °C). A dual-cure composite resin (Core Paste XP) was selected to build-up the core around posts, obtaining cylindrical specimens that were serially cut in beams and subsequently loaded in tension (μTBS) at a cross-head speed of 1 mm/min until failure. Bond strength data were statistically analyzed by two-way ANOVA and Student–Newman–Keuls tests ($\alpha = 0.05$).
Results. Warm air-drying determined significantly higher bond strengths ($p < 0.001$) for glycid-oxi-propyl-trimetil-oxi-silane (11.6 MPa) and 4-methacryolxyethyl trimellitate anhydride/γ-trimethoxysilyil propyl methacrylate silane (11.7 MPa). These two systems exhibited lower bond strengths (6.9 and 8.8 MPa, respectively) than 3-methacryloxypropyltrimethoxysilane (11.0 MPa) when dried at 21 °C. No statistical differences were recorded for 3-methacryloxypropyltrimethoxysilane when drying at 21 or 38 °C.
Significance. The composition of the silane coupling agent in terms of acidic content, solvent rate or degree of hydrolysis may influence resin/post bond strength when dried at 21 °C. Drying at 38 °C most likely facilitates the evaporation of solvents present in the silane agent, resulting in increased bond strength of the composite resin to the fiber post.

1. Introduction

Several studies suggested the use of silane coupling agents in coating applications to promote adhesion between inorganic surfaces and polymeric molecules [1,2].

Organosilanes have the formula R′-Si-$(OR)_3$ with an organic functional group (R′) and three alkoxy groups (R): the chemical reaction begins with the hydrolysis of the alkoxide groups (R) into silanols (SiOH) that may condense forming siloxane bonds [2,3].

Many factors (pH, presence of solvents, molecule size, etc.) may exert an influence in the way silane molecules can absorb, condense or interact with the substrate, influencing coupling effectiveness [4,5].

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0109-5641/$ – see front matter © 2005 Academy of Dental Materials. Published by Elsevier Ltd. All rights reserved.
doi:10.1016/j.dental.2005.11.024

To accelerate the mechanism of chemical interaction between the silane and the inorganic surface, the reaction may be catalyzed by acid treatment or heating [6,7]. Heat treatment of silanated glass is routinely performed in the glass industry to maximize bond strength [8]. Silane has been proven to increase ceramic-composite bond strength during luting procedures or when repairing chipped ceramic restorations [9–11]. Drying with hot air increases the effectiveness of some silane coupling agents when bonding ceramics to composite resins [7,12].

High temperature silane heat treatment (70–80 °C) is not feasible for chair-side procedures, but a stream of warm air (38 °C) may be used for this purpose [7].

Fibre posts are extensively used in combination with composite resins to directly restore endodontically treated teeth [13,14]. The efficacy of silane coupling agents increasing bond strength between fiber post and composite core restorations have been recently reported [15–18]. However, no information is available concerning the possible influence of different silane coupling agents' composition or silanizing modalities on post/composite bond strength. In particular, the possible influence of heating on the condensation reaction of silane molecules on the post surface is still unknown. The aim of the study was to determine the effect of warm air drying and different silane coupling agents on the achieved bond strength between fiber posts and resin composite.

The null hypothesis is that silane composition and air-drying temperature do not influence the microtensile bond strength between fiber post and composite resin.

2. Materials and methods

Thirty quartz fiber posts, with a maximum diameter of 1.80 mm in the cylindrical coronal portion and 1.0 mm at the radicular end (DT Light Post #2, batch no.100US0311A, RTD, St. Egéve, France) were used for this study. DT Light posts are made of unidirectional pre-tensed quartz fibers (60%) bound in an epoxy resin matrix (40%).

The posts were etched in 10 vol% hydrogen peroxide solution (Panreac Química, Barcelona, Spain) for 20 min at room temperature [19]. They were rinsed with tap water and ultrasonically cleaned for 10 min in deionised water (P Selecta S.A. Abrera, Barcelona, Spain), subsequently immersed in 96% ethanol and dried with an air stream.

Six experimental groups ($n = 5$) were formed and three different silane coupling agents were used: a pre-hydrolyzed silane coupling agent containing 3-methacryloxypropyl-trimethoxysilane (3-MPS) (Monobond-S, batch no. E53184, Ivoclar-Vivadent, Schaan, Liechtenstein); a two-component silane coupling agent containing 4-methacryolxyethyl trimellitate anhydride (4-META) and trimethoxysilyil propyl methacrylate (γ-MPTS) (Porcelain Liner M, batch no. GF1, Sun Medical Co. Ltd., Japan); a pre-hydrolyzed silane coupling agent containing glycid-oxi-propyl-trimetil-oxi-silane (GPS) (Porcelain Silane, batch no. 4101PFS, BJM Lab, Or-Yenuda, Israel) at two different air-drying temperatures (21 and 38 °C). The tested materials were applied following manufacturers' recommendations. The composition and application mode of the tested materials are described in Table 1.

pH measurements were performed for all tested silane coupling agents with a digital pH-meter and a glass electrode calibrated with standard buffer solutions (Micro pH 2000, Crison Instruments, Alella, Spain).

After etching and silanizing the post surface, composite build-up was performed following a technique previously described by Goracci et al. [15] and using a dual-curing resin composite (Core Paste XP, batch no. 030653101, Dent Mat, Santa Maria, CA, USA). Core Paste XP is a low viscosity core material and contains glass fillers in a methacrylate matrix. Samples were stored 24 h at room temperature before testing.

Microtensile test specimens were prepared by sectioning each sample with a diamond saw under water cooling (Isomet 4000, Buehler, Lake Bluff, IL, U.S.A). A medium of 29 beams of 1-mm in thickness were tested for each group. For the microtensile bond strength test, each beam was glued with cianoacrylate (Zapit, Dental Ventures of America, Corona, USA) to the flat grip of a testing device (Bencor, Multi-T, Danville Engineering, San Ramon, CA, U.S.A.) and loaded in tension at a cross-head speed of 1 mm/min until failure (Instron Model 4411, Instrom, Canton, MA, U.S.A.). The modes of failure were evaluated after testing under a stereomicroscope (Olympus SZ-CTV, Olympus, Tokyo, Japan) at 40× magnification. Failure modes were classified as adhesive (at the post/core interface), cohesive (within the resin composite) or mixed (combination of the two modes on the same surface).

Interfacial bond strength values were expressed in MPa using a mathematical formula previously described by Bouillaguet et al. [20]. Data were analyzed by two-way ANOVA

Table 1 – Silane coupling agent compositions and procedures tested in the study

Material	Composition	Application
Monobond-S (Ivoclar-Vivadent, Schaan, Liechtenstein), pH ≈ 3.8	3-MPS (1%), ethanol/water-based solvent, acetic acid	Apply with a brush; leave undisturbed for 60 s; gently air-dry
Porcelain Liner M (Sun Medical Co. Ltd., Japan), pH ≈ 4.5	Liquid A: MMA, 4-META (10%); liquid B: MMA, γ-MPTS (10%)	Apply with a brush; gently air-dry
Porcelain Silane (BJM Lab, Or-Yenuda, Israel), pH ≈ 1.8	GPS (3%), ethanol-based solvent	Apply with a brush; gently air-dry

3-MPS: 3-Methacryloxypropyltrimethoxysilane; MMA: methyl methacrylate; 4-META: 4-methacryolxyethyl trimellitate anhydride; γ-MPTS: trimethoxysilyil propyl methacrylate; GPS: glycid-oxi-propyl-trimetil-oxi-silane.