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ORIGINAL RESEARCH

Influence of silane and solvated bonding agents on the bond strength to glass-fibre posts

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Abstract

The combined use of silane and solvated bonding agents on the bond strength to glass-fibre posts was investigated. A model Bis-GMA/HEMA adhesive was formulated with no solvent, 30% of ethanol or 80% of acetone. The surfaces of rectangular-shaped posts were silanated or not and one of the agents was applied, except for the control group. Cylinders of resin cement (RelyX ARC, 3M ESPE, Saint Paul, MN, USA) were built-up on the surfaces ($n = 20$) and submitted to shear testing. All groups showed higher bond strengths when the surfaces were silanated. When no silanization was carried out, the use of bonding agents, either solvated or non-solvated, increased the bond strengths. All groups treated with both silane and bonding agent showed higher bond strengths than the group that was only silanated. Control and ethanol-based adhesives were similar, whereas the acetone-based agent yielded higher bond strengths. Adhesive failures were predominant. Combination of silane and adhesive enhanced the bond to fibre posts.

Introduction

A controversial issue regarding the adhesive cementation of glass-fibre posts is the use of silane coupling agents. Silanes are bifunctional molecules with one end of the molecule capable of reacting with inorganic glass fibre and the other with organic resin. Previous studies reported contrasting results regarding the effectiveness of silanes in enhancing the bonding of resin cements to fibre posts (1–5). Another issue that raises controversy is the use of a polymerisable bonding agent after silane. As the silane forms a very thin layer over the surface, the use of a bonding agent could provide a better mechanical keying with the post.

When using a solvated bonding agent over silane, there might be an effect of the solvent (ethanol or acetone) with the silane agent. At least theoretically, non-solvated adhesives should be used. Few studies have evaluated the effect of commercial bonding agents on the bond strength to fibre posts (6,7). As not only the solvent type but also the comonomer formulation changes among proprietary adhesives (8), it is difficult to compare the bond strengths of resin cements to fibre posts when different commercial bonding agents are used.

The aim of this study was to evaluate the bond strength of a dual-cured resin cement to glass-fibre posts treated or not with a silane coupling agent and with model bonding agents containing either acetone, ethanol or no solvent. The hypotheses tested were that both (i) the silane treatment and (ii) the bonding agents would enhance the bond strength to the glass-fibre posts.

Materials and methods

Formulation of the experimental bonding agents

A model (di)methacrylate comonomer blend was obtained by mixing bisphenol-A glycidyl dimethacrylate (Evonik, Essen, Germany) and hydroxyethyl methacrylate (Sigma-Aldrich, St. Louis, MO, USA) at a 3:2 mass ratio. A mass fraction of 0.4% of camphorquinone (Esstech, Essington, PA, USA) and 0.8% of ethyl-4

Table 1 Groups tested and means (standard deviations) for bond strength, MPa ($n = 20$)

	Bonding agent			
Silane	None	Non-solvated	Ethanol-based	Acetone-based
No	3.4 (1.6)^{C,b}	6.0 (1.6)^{B,b}	5.4 (1.4)^{B,b}	9.2 (2.1)^{A,b}
Yes	9.6 (2.5)^{C,a}	13.1 (2.5)^{B,a}	14.6 (2.2)^{B,a}	17.9 (2.8)^{A,a}

Distinct capital letters in a same line indicate differences for bonding agents; distinct lowercase letters in a same column indicate differences for silane application ($P < 0.05$).

dimethylamino benzoate (Sigma-Aldrich) were included to make the blend photocurable. From the model blend, two solvated bonding solutions were obtained by mixing the resin with 30% or 80% mass fractions of absolute ethanol or acetone, respectively. The concentration of solvents was defined based on a previous investigation testing commercial two-step, etch-and-rinse adhesives (9). Non-solvated resin was also tested.

Preparation of specimens

The conventional dual-cured resin cement RelyX ARC (3M ESPE, Saint Paul, MN, USA) was tested. Customised rectangular (6 × 5 mm, 2 mm thick) glass fibre-reinforced epoxy resin posts were obtained from Angelus (Londrina, PR, Brazil) and embedded in epoxy resin. The bar geometry was important to allow obtaining specimens on a flat post surface. The specimens were ultrasonically cleansed in distilled water for 10 min and dried with compressed air. The posts were either treated or not with silane coupling agent (Angelus) and then with the different bonding solutions, as shown in Table 1. After treating the post surface, the solvent (for the bonding agent or silane) was evaporated with oil-free compressed air for 10 s, even for the non-solvated agent. The adhesive was light-activated for 20 s using a light-emitting diode unit (Radii; SDI, Bayswater, Victoria, Australia) with 600 mW cm^{-2} irradiance.

The experimental setup used to obtain specimens for the bond strength test was described elsewhere (10). Briefly, 0.5 mm thick elastomer molds with a cylindrical orifice (diameter 1.2 mm) were placed onto the post surfaces and held tightly. Equal volumes of base and catalyst pastes of the cement were mixed for 10 s; the orifices were filled with the resin cement; and the molds covered with a polyester strip and a glass slide. The samples were submitted to a constant and uniform 500 g cementation load for 3 min and the resin cement photoactivated for 30 s. For each group, 20 specimens were prepared.

Bond strength test and failure analysis

The samples were stored in distilled water at 37°C, for 24 h. For the shear bond test, a thin steel wire (diameter 0.2 mm) was looped around each cylinder and aligned with the bonding interface. The test was conducted in a mechanical testing machine (DL500; EMIC, São José dos Pinhais, PR, Brazil), at a cross head speed of 0.5 mm min^{-1} until failure. Bond strength values were calculated in MPa, and data were submitted to two-way ANOVA. All pairwise multiple comparison procedures were performed by the Student-Newman-Keuls' method ($P < 0.05$). The fractured specimens were examined under optical microscopy at a 40× magnification. Modes of failure were classified as adhesive failure or mixed failure (remnants of cement and/or bonding agent on the post surface).

Scanning electron microscopy (SEM) analysis of the bonded interfaces

In order to observe the morphology of the bonded interfaces, additional specimens were obtained and embedded cross-sectionally in epoxy resin, similar to what has been previously described (11). The specimens were wet-polished with 600-, 1200-, 1500-, 2000- and 2500-grit SiC papers followed by polishing with 3, 1, 0.25 and 0.1 μm diamond polishing suspensions. The specimens were coated with gold and the cross-section profiles examined by SEM (LEO 435 VP; LEO Electron Microscopy, Cambridge, UK) at 20 kV.

Results

Results for the bond strength test are shown in Table 1. The statistical analysis showed the factors 'silane application' and 'bonding agent' were both significant ($P < 0.001$), as well was their interaction ($P = 0.005$). All groups showed significantly higher bond strengths when the surfaces were treated with silane ($P < 0.001$), irrespective of the use of any of the bonding agents. When silanization of the post surface was not carried out, the use of a bonding agent, either solvated or non-solvated, also significantly increased the bond strength to the glass-fibre posts ($P \leq 0.003$).

For the combined use of silane and adhesive, all groups treated with bonding agent presented significantly higher bond strengths than the group that was only silanated ($P < 0.001$). Comparing the bonding agents, the non-solvated and ethanol-based adhesives presented similar results, whereas the acetone-based material yielded significantly higher bond strengths than the other bonding resins ($P < 0.001$), regardless of using silane or not. Dis-

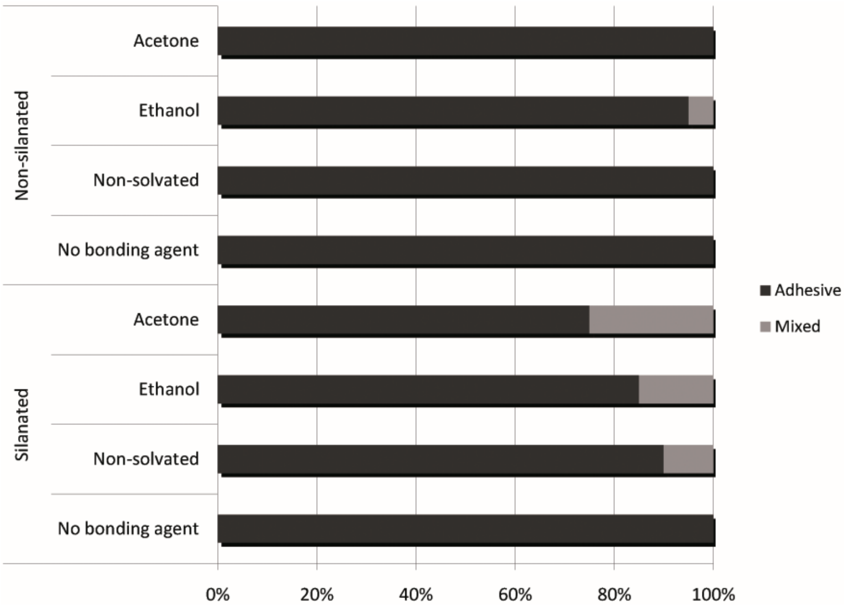

Figure 1 Distribution of failure modes among groups. A predominance of adhesive failures was detected for all groups. The number of mixed failures was higher for the silanated groups.

tribution of failure modes is presented in Figure 1. A predominance of adhesive failures was detected for all groups, irrespective of the use of silane and/or bonding agent. The number of mixed failures was, however, higher for the silanated groups.

Representative SEM pictures of the bonded interfaces are shown in Figure 2. No appreciable differences were observed regarding the homogeneity and thickness of the adhesive layer formed by either of the solvated bonding agents compared with each other, or compared with silane alone. On the other hand, the non-solvated agent formed a thicker adhesive layer between the post and resin cement compared with all other groups.

Discussion

Application of silane enhanced the bond strength to the posts, confirming the first hypothesis. Organosilanes bond to the silica in the glass fibres through the formation of siloxane bonds after hydrolysis of the alkoxy groups into silanol groups (12,13). Improvement in the surface wettability is another effect of silanization. As an intimate contact between the interfacing materials is established, van der Waals' forces may become effective providing a physical adhesion, contributing to the chemical reaction (14). The resin phase of the cement then copolymerises with the methacrylate group of the silane, increasing the bond strengths. A recent study has indicated, however, that care should be taken when associating silanes and self-adhesive resin cements (10).

The use of bonding agents alone, either solvated or non-solvated, also increased the bond strengths. This finding indicates that not only chemical reactions (as in the case of silane application) but also mechanical interlocking with the post surface may have a role in improving the bonds. This effect is evident when observing that the combined use of silane and any of the adhesives generated higher bond strength than the use of silane alone. This result is explained by the summation of all events in the treated surfaces enhancing the bond of the resin cement: chemical linkage through siloxane bonds, better surface wetting and better mechanical keying. This is evidence of the positive effect of applying a bonding agent after silane. Therefore, the second hypothesis is also confirmed.

The use of model adhesive agents with the same comonomer composition allowed comparisons restricted to the solvents themselves. While the non-solvated and ethanol-based agents presented similar results, the acetone-based adhesive yielded the highest bond strengths. One could expect that the lower resin content in the acetone-based agent could generate a thinner, more homogeneous layer on the post surface, with less porosity. However, this effect was not observed in the SEM analysis. A proposed explanation for the better bonding of the acetone-based agent is that the high

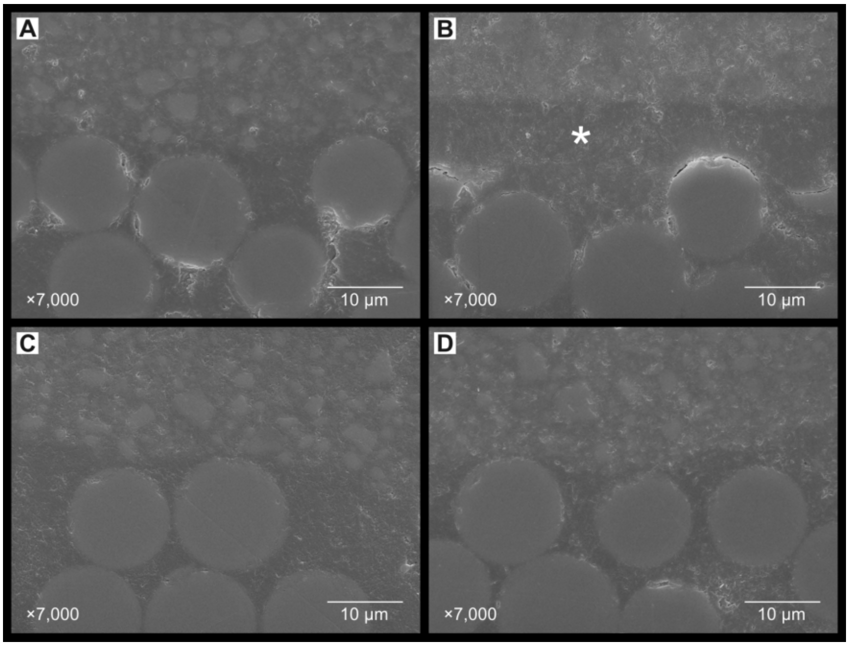

Figure 2 Representative SEM images of the bonded interfaces (A = only silane; B = silane + non-solvated bonding agent; C = silane + ethanol-based agent; D = silane + acetone-based agent). The non-solvated agent formed a thick adhesive layer between the post and resin cement (asterisk on B), whereas no appreciable differences were observed for the solvated bonding agents compared with silane alone.

solvent content (80%) could have slightly dissolved the epoxy resin on the post, roughening the surface and enhancing the mechanical interlocking. The lower solvent content on the ethanol-based agent (30%) might explain why this adhesive had a similar result compared with the non-solvated material. In addition, as the acetone content may vary among commercial bonding agents, the effect of surface dissolution is expected to be material-dependent.

The present results show that the combined use of silane and adhesive agent, especially the acetone-based material, has a significant effect in increasing the bond strength of conventional dual-cure resin cements to glass-fibre posts. In addition, the present study indicates that any bonding agent, either solvated or non-solvated, may provide a better bond compared with silane alone. It is difficult to predict, however, whether this enhanced bond would have a significant impact on the clinical performance of adhesively luted posts. That notwithstanding, it is of clinical interest to have a long-lasting bond of all components involved in the restoration of endodontically treated teeth. Therefore, association of silane and bonding agents might be encouraged in the clinical practice. Future studies should focus on long-term performance of glass-fibre posts bonded to root canals using a combination of silane and adhesive agent.

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